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APOLLO 14 COMPOSITE CASTING DEMONSTRATION FINAL REPORT

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16. ABSTRACT

The results of the Apollo 14 Composite Casting Demonstration are presented in this report.

The purpose of the demonstration was to show that mixtures of materials of different specific gravities would remain stable in the liquid state and during freezing in low g and not segregate as they do on earth.

To demonstrate this effect an inflight demonstration was performed on the Apollo 14 mission during the translunar and transearth coast periods. The apparatus consisted of an electrical heater, a heat sink device for cooling, and sealed metal capsules containing matrix materials haveing a low-melting point and dispersants. These were selected as models of potentially more useful materials. During the time available the astronauts processed 11 samples.

The evaluation of the demonstration samples was accomplished by comparing space processed (flight) samples with (control) samples processed on the ground under otherwise similar conditions. In the low-g environment of space flight the dispersions of particles, fibers, and gases in a liquid metal matrix were maintained during solidification. Dispersions of normally immiscible liquids were also maintained during solidification.

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APOLLO 14 COMPOSITE CASTING DEMONSTRATION FINAL REPORT

SUMMARY

The results of the Apollo 14 Composite Casting Demonstration are presented in this report.

On earth, materials of different specific gravities normally segregate from a mixture when at least one of the components of the mixture attains the liquid state. The segregated structure may then be seen in the resolidified material. It has been postulated that such mixtures, in the very low-g environment of space flight, would remain stable in the liquid state and during freezing so that novel material structures can be made.

To demonstrate this effect a composite casting demonstration was performed on the Apollo 14 mission during the translunar and transearth coast periods. The apparatus was simple and light. It consisted of an electric heater with a controlled maximum internal temperature of 385.93°K (235° F) and of a heat sink device for cooling. The 11 samples were contained in standard sealed cartridges and were processed sequentially by the astronauts. Some of the materials in the cartridges had been premixed, others had to be mixed after melting by a prescribed method of manual shaking. The materials were carefully chosen after extended studies; they were intended to model the reduced gravity effects which would be expected to occur in other more directly useful materials. The choice of these materials and the heater design were dictated primarily by considerations of simplicity of operation, weight, power consumption, maximum touch temperature, toxicity, flammability, availability of prior applicable knowledge about the metal system, and simplicity of preparation as a flight sample.

Owing to the weight and astronaut time limitations, only one sample of each composition was flown.

The evaluation of the demonstration samples was performed by comparing space processed (flight) samples with (control) samples processed on the ground under otherwise similar conditions. The evaluations were performed using radiography, macroscopic and microscopic analysis of the

surface features and of sections, analysis of fractures, density determination, and other customary laboratory techniques as well as statistical and theoretical analyses. The evaluation procedures as well as the processing methods were developed on early "development samples".

The results obtained from the evaluation of three sets of samples may be summarized as follows:

1. Premixed Samples. The samples were precompressed metal powder compacts containing 30 percent (vol) of dispersed non-melting particles. In one pair (flight and control samples) a more uniform distribution of particles was observed in the flight sample. The results from the second pair of samples proved inconclusive because only partial melting had taken place in the flight sample. In both pairs, distinct differences in surface features were observed between control and flight samples apparently owing to the predominant influence of surface tension effects under reduced gravity.

2. Samples with Fibers or Particles and/or Gas Added to Matrix. These samples were hand-shaken for mixing. From a comparison of the control and the flight samples the following conclusions were reached:

The flight samples had markedly enhanced dispersion and uniformity of distribution of gases and non-melting dispersants even though, in some instances, the dispersant had non-wetting characteristics; the use of a dispersant appears to enhance the retention of gases; there were marked differences in surface features between control and flight samples – apparently the flight samples tended to "free float" within the capsules and hence assume shapes dictated by surface tension effects.

3. Samples with Two and Three Phase Immiscible Mixtures. (The third phase, when present, was gas or tungsten spheres.) These samples were mixed by manual shaking after melting. The following results were observed when comparing the flight with the control samples:

a. Stable dispersions exist in all three flight samples.

b. The dispersions range from very fine to relatively coarse.

c. All such dispersions are unstable on earth and hence are not observed in the control samples.

d. The dispersions were not homogeneous: this is attributed to the relatively primitive mixing procedure by hand shaking and to the thermal gradients existing during solidification.

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4. General Observations on All Demonstrations: Owing to the "free float" condition under low-g conditions, the thermal contact of the melt with the container was intermittent and uncontrolled. Hence the melting and particularly the solidification processes were not the same for the flight and the control samples. This is evidenced by unusual metallographic structures in several flight samples.

In the detailed reports from the investigators, recommendations for new or improved experiments are made to answer questions raised by the present demonstrations. Conclusions are also drawn as to the significance of these demonstrations for the Space Manufacturing program.

INTRODUCTION

The use of the weightless environment of space flight for manufacturing unique materials and products was suggested several years ago[1].

Presuming the absence of buoyancy and of thermal convection in zero g it was postulated that materials of different densities mixed in the liquid state would not segregate and that such a dispersion could be maintained during cooling and solidification[2]. This report describes demonstrations of this effect using simple apparatus, and materials which are intended as models only.

The purpose of the composite casting demonstration was to obtain preliminary data on the processes of melting, mixing, and solidification of composite materials. Composite casting is defined as the casting of a material from a mixture of a liquid matrix and solid reinforcements such as whiskers, particles, or fibers. A variation of composite casting is obtained when gas is added to form foam material (which may also be reinforced by whiskers, etc., if desired). Another variation is obtained when normally immiscible liquid materials are dispersed one in the other and solidified.

The composite casting process had been identified in a previous study [1] as a process which made the most direct application of the presumed zero-g phenomena and one where an early demonstration could be performed.

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1. National Aeronautics and Space Administration , Manufacturing Technology Unique to Zero-Gravity Environment, Marshall Space Flight Center Meeting, Huntsville, Alabama, November 1968.

2. National Aeronautics and Space Administration, Space Processing and Manufacturing, Marshall Space Flight Center Meeting, Huntsville, Alabama, October 21, 1969.

The data presented in this report were derived from a comparison of samples processed in space with those processed on earth under otherwise similar conditions.

The extended zero g available during the transearth coasting phase of an Apollo mission was seen as an opportunity to give an early demonstration of the postulated effects on materials processing in an environment where gravity is reduced to a very small value.

Approval was given in September 1970 to proceed with the design and development of an in-flight demonstration of composite casting and analysis for compatibility with Apollo 14.

The demonstration apparatus was designed and developed by the Process Engineering Laboratory, Marshall Space Flight Center (MSFC), with consulting help on selection of sample materials provided by General Dynamics Convair Division and A. D. Little, Inc., under contracts NAS8-24979 and NAS8-26637 respectively. Other contributors to the selection of samples were Astronautics Laboratory, Space Sciences Laboratory, and Research Planning Office at MSFC, and Cornell Aeronautical Laboratory and TRW Systems Group. The Quality and Reliability Laboratory performed all qualification and acceptance tests with the support of Astronautics Laboratory for flammability and shock tests. All required reviews, plans, and documentation were accomplished and approved and the hardware fabricated and delivered to Kennedy Space Center by December 10, 1970.

DEMONSTRATION APPARATUS AND PROCEDURES

The apparatus consisted of an electrical heater, a storage box for the heater which also served as a heat sink for cooling the samples to touch temperature before removal, and 18 samples contained in hermetically sealed metal capsules. A beta cloth bag resembling a cartridge belt was used to store the 18 sample capsules.

Procedures called for inserting each capsule into the heater as shown in Figure 1, heating for a prescribed time to melt the capsule contents, shaking in some cases to mix the materials, and cooling by placing the heater and capsule onto the heat sink as shown in Figure 2. The right half of the aluminum storage box was a massive section of aluminum with an integrally machined heat sink pin which made contact with the specimen capsule. A
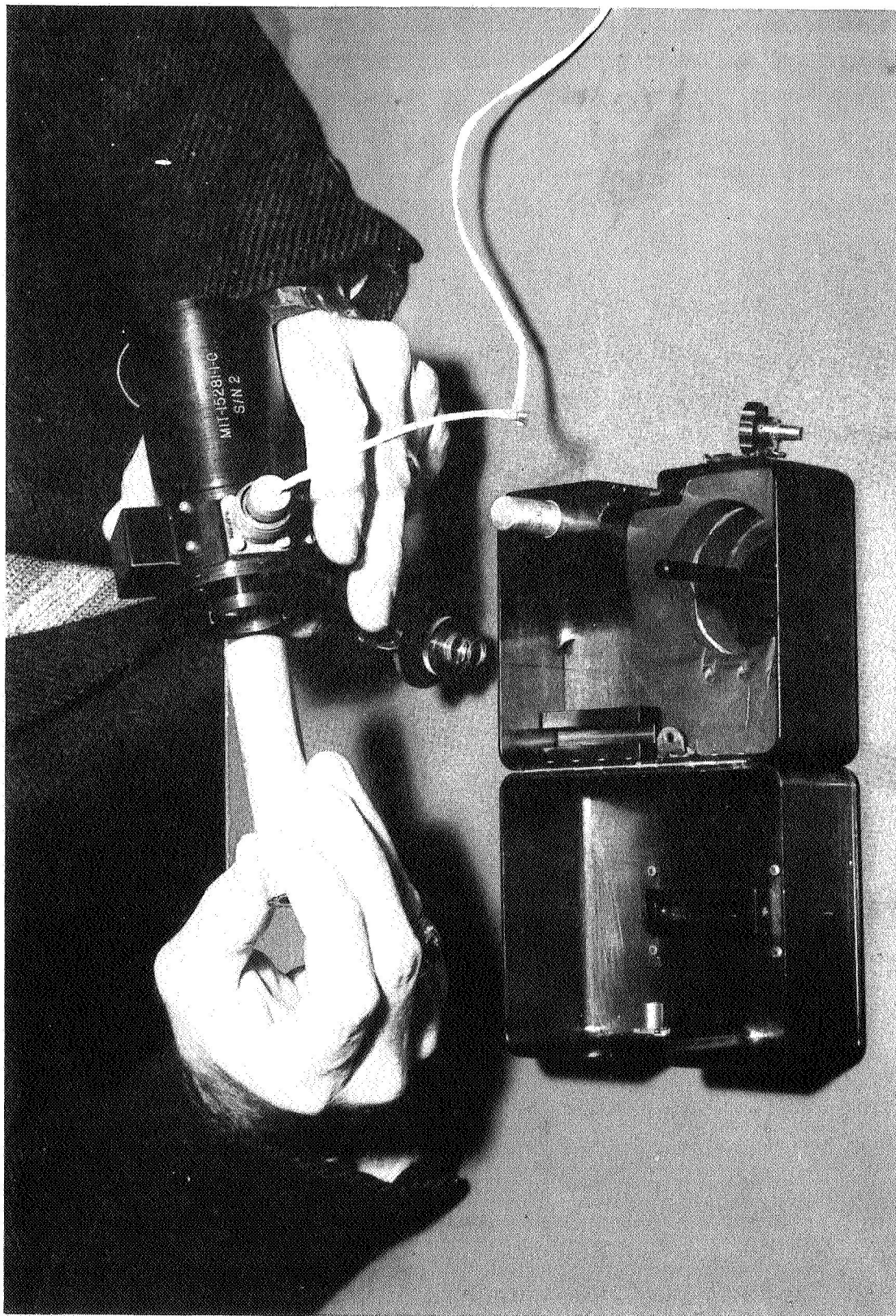
Figure 1. Composite casting heater and box with typical specimen capsule.
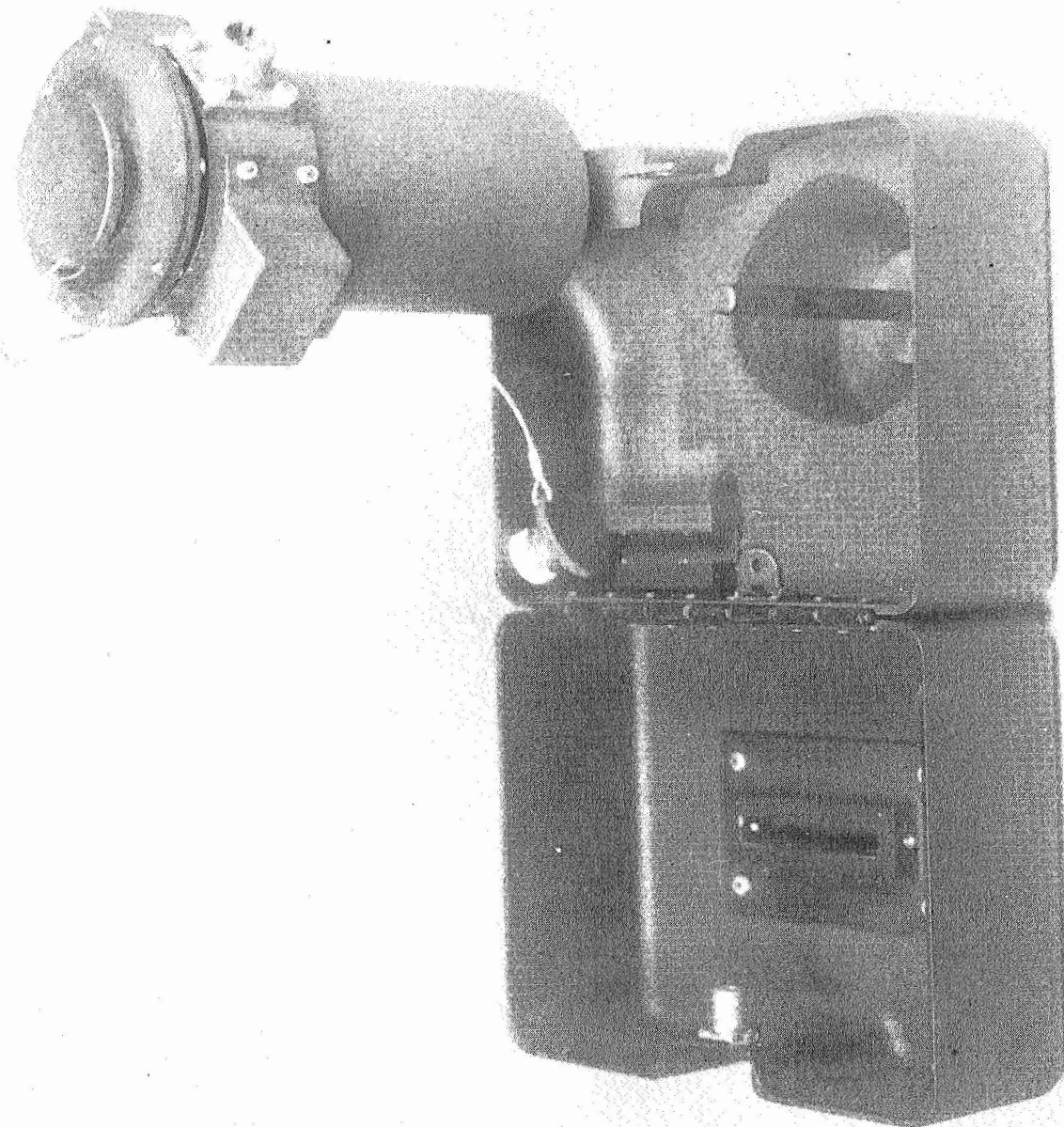
Figure 2. Composite casting heater on heat sink.

better view of the heat sink pin is seen in Figure 1. A bayonet type connection was made between the heater and the pin; the spring in the upper cap held the capsule tightly against the heat sink.

The heater operated on 27.5 V, DC from the Command Module power bus using the Data Acquisition Camera power cable. Connection was made at a utility receptacle, and a switch on the panel was used to switch the heater on and off. Nominal power consumption for the heater was 34 W. A cross section of the heater with a sample capsule inside is shown in Figure 3. Redundant thermal switches were installed to insure that the outside surfaces of the heater did not exceed 313.65°K (105°F). The maximum temperature reached by the capsule after 10 min of heating was approximately 385.95°K (235°F). Cooling times of no less than 30 min on the heat sink reduced the temperature of the capsules to less than 310.95°K (100°F).

An abbreviated form of the procedures followed by the astronauts is shown in Figure 4. Where applicable, the shaking procedure was as follows:

The shaking procedure used after melting the samples involved bumping each end of the heater against the heel of the hand 4 times to dislodge any particles trapped in the ends of the capsule; 3 cycles of alternately shaking the heater axially 10 times, oscillating in a rotary motion 15 times; and finishing by oscillating 10 times going from a vigorous motion to very slow. Tests with model systems in transparent models showed this to be the best mixing procedure under the circumstances. A mechanical device would have been preferred to obtain a more vigorous, controlled mixing; however, the development and integration of such a device into the demonstration was not possible in the time available.

During translunar and transearth coasts, 11 of the 18 samples were processed. Sample No. 10 was heated at least twice because Reaction Control System (RCS) firings occurred during the first cooling cycle. Several RCS firings occurred at approximately 15 min into the cooling cycle on Sample No. 12; however, the sample was not reheated because of mission constraints. The astronauts reported that the heater performed well and that there were no problems with the procedures. Postflight tests of the heater showed that its performance was still within specifications.

The sample capsules and heater were held in quarantine with the Command Module until April 5, 1971, when they were removed and returned to MSFC for evaluation.
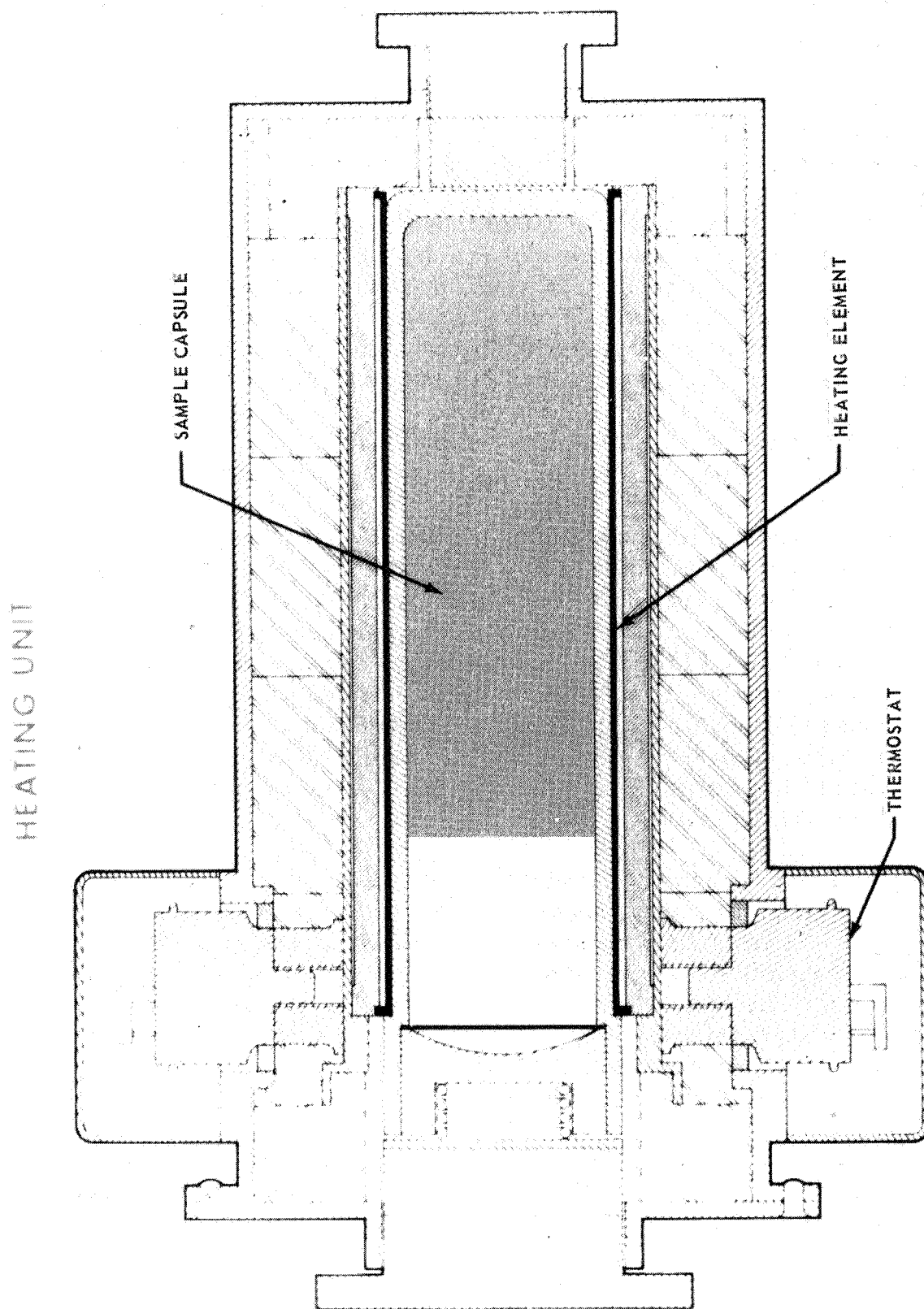
Figure 3. Cross section of heating unit with sample capsule.

Timer — A5
16mm power cable — B3
Composite specimen & heater box —A8
Snap specimen below B5 & B6
Open heater box, lock lid
Mount box in tunnel w/spring
PNL 100 pwr. sw. — OFF
Install pwr. cable to heater & PNL 100
Install extractor pin
Perform following procedure for the 18 specimen listed below
1 Remove specimen & install in heater
Place heater in box
Set timer — X min for heating
PNL 100 pwr. sw. — ON
After X min — PNL 100 pwr. sw. — OFF, Remove Pwr. Cable from Heater
Remove heater & SHAKE/NO SHAKE
Shake procedure for specimen 4 thru 14 consists of the following:
    a. Bump against heel of hand 4 times each end alternately
    b. Shake linearly 10 cycles & oscillate 15 cycles
    c. Repeat (b) 3 times
    d. Oscillate 10 cycles going from vigorously to very slow
Place heater on heat sink
Set timer X min for cooling
NOTE: If an RCS firing occurs during cooling cycle on specimen 4 thru 14
      repeat heating
After X min remove heater & remove specimen using extractor pin
Restow specimen — indicate below specimen was tested
Repeat at step 1 for all specimen

SPECIMEN NUMBER	HEATING TIME (min)	SHAKE/ NO SHAKE	COOLING TIME (min)	COMMENTS	SPECIMEN COMPLETED (GET)	
					(hrs)	(min)
1	10	No Shake	30		201	10
2	10	No Shake	30		201	55
3	10	No Shake	30			
4	10	Shake	30		054	35
5	10	Shake	30		057	17
6	10	Shake	30	Contains Pellet	058	13
7	10	Shake	30		177	46
8	10	Shake	30		178	41
9	10	Shake	30	Contains Pellet	179	29
10	10	Shake	30	Contains Pellet	187	30
11	10	Shake	30		190	40
12	10	Shake	30	*Contains Pellet	193	02
13	10	Shake	30			
14	10	Shake	30			
15	13	No Shake	30			
16	8	No Shake	30			
17	13	No Shake	30			
18	13	Do Not Touch	120			

*A small number of jet firings occurred after 15 minute cooling time.

Figure 4. Metal Composite Procedures (From Apollo 14 Flight Log).

COMPOSITE MATERIALS SELECTION AND SAMPLE PREPARATION

The selection of materials for the demonstration was limited to low melting point materials because of the constraints on power consumption, weight and size of apparatus, and of safety considerations. All sample materials were required to meet Apollo specifications for toxicity, odor, and flammability although they were sealed in metal capsules.

Two types of matrix materials were considered for the demonstration: (1) a metal or alloy and (2) a transparent or translucent inorganic or organic material which would serve as a model for a crystalline metal matrix. Both types of matrix material were required to have a melting temperature below 349.85° K (170° F) and above 313.65° K (105°F). Evaluation of a variety of matrix materials and reinforcements was carried out concurrently inhouse and by contractors. A. D. Little, Inc. was engaged for the evaluation of candidate nonmetallic materials, and General Dynamics/ Convair for the evaluation of candidate metallic materials. Inhouse investigations using primarily metallic materials were directed mainly towards the development of processing techniques.

Evaluation of Candidate Nonmetallic Matrix Materials

A. D. Little considered several types of materials as candidates for the matrix including inorganic salt hydrates, salt eutectics, fluorocarbons, organic phosphates and silicates, and low-melting organics. The principal criteria for selection of materials were:

a. Melting point below 349.85°K (170° F).

b. Low toxicity.

c. Low flammability.

d. Low vapor pressure.

e. Readily available (preferably flight approved materials).

f. Good model for crystalline metal matrix.

g. Transparent, if possible.

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h. Significant density difference between matrix and reinforcing materials.

i. Wetting of reinforcing materials by matrix.

j. Chemical and physical compatibility of matrix material, reinforcing material and container.

After an initial screening of the candidate matrix materials, A. D. Little selected cobalt nitrate, tri-p-tolylphosphate, decafluorobiphenyl, and benzophenone for laboratory screening tests. A list of the materials screened and the details of the screening tests performed on the four candidates are reported in Reference 2. On the basis of their screening tests, cobalt nitrate was selected as the most acceptable material and decafluorobiphenyl as the next most promising. The melting points of cobalt nitrate and decafluoro-biphenyl are 330.35°K (135°F) and 341.45°K (155°F) respectively.

In addition to their investigations of metallic matrix materials, General Dynamics/Convair also considered nonmetallic materials for a transparent matrix. They screened a number of candidate materials and recommended Burtonite 44, a saccharide, as a candidate. Details of their evaluation are reported in Reference 5. A solution of 1.5 percent Burtonite 44 in water has a melting temperature of 319.25°K (115°F) and a density of 1.0 g/cm^3. It is transparent as a solid and a liquid. The primary disadvantage of the Burtonite solution was the danger of boiling and hence of increased pressure within the specimen capsule when the temperature reaches 373.15°K (212°F).

In the final selection of a nonmetallic matrix material, cobalt nitrate and decafluorobiphenyl were ruled out because of lack of time to complete toxicity and flammability tests. Burtonite 44 was ruled out because of its low boiling temperature and the difficulty in handling the jelly-like material. Paraffin, which had been recommended for the immiscible materials demonstrations, was finally selected although it did not satisfy the criteria of transparency and has a high rate of shrinkage upon solidification. It is translucent and provides more visibility of the dispersed reinforcing materials than the metallic matrix materials.

Evaluation of Candidate Metallic Matrix Materials

Initially investigations were limited to a group of bismuth-based eutectic alloys made by Cerro Copper and Brass Company. Those considered were:

Trade Name	Constituents					Melting Point	
	Bi	Pb	Sn	Cd	In	°K	°F
Cerrolow 117	44.7	22.6	8.3	5.3	19.1	320.45	(117)
Cerrolow 136	49.0	18.0	12.0	--	21.0	330.85	(136)
Cerrobend	50.0	26.7	13.3	10.0	--	343.15	(158)
Cerro Solder	(Automatic Sprinkler Spec.)					346.95	(165)

Preliminary screening tests were performed by MSFC and Convair. Cerrobend was selected for further laboratory experiments because it offered good strength and creep resistance at room temperature, an acceptable melting temperature, and high density compared with the reinforcing materials being considered. Subsequently Cerrobend was eliminated as a matrix material because of the toxicity of the cadmium it contains. A 66 percent indium, 34 percent bismuth eutectic alloy was substituted. In-Bi has a density of 8.0 g/cm^3 and a melting point of 345.15°K (162°F). In-Bi has characteristics similar to those of Cerrobend except for its strength. General Dynamics/Convair reported the following data [3]:

Alloy	Test Temperature		UTS
	°K	°F	
Cerrobend	297.15	(100.8)	2407
	273.15	(32.0)	6012
	195.15	(-108.4)	7509
In-Bi	298.15	(77.0)	1261
	273.15	(32.0)	2293

Evaluation of Candidate Reinforcement Materials

Evaluations of a variety of reinforcement materials were carried out principally inhouse and by General Dynamics/Convair. Limited investigations of reinforcement materials were made by A. D. Little. The candidate materials included:

a. Chopped metal wires.

b. Chopped glass filaments.

c. Chopped boron filaments.

d. Whiskers.

e. Metallic particles.

f. Oxide particles.

The criteria for selection of reinforcement materials and surface treatments were:

a. Chemical compatibility with matrix.

b. Wetting by the liquid matrix.

c. Adherence of the solid matrix to the reinforcement.

d. High bond strength between reinforcement and solidified matrix.

e. High reinforcement material strength relative to matrix.

f. Significant density difference between matrix and reinforcement.

Initially experiments were conducted with the Cerrobend matrix and a selection of most reinforcements was a result of those experiments. After the matrix was changed to In-Bi, additional tests were made to verify the selections.

The metallic wires tested were copper, stainless steel, carbon steel, galvanized steel, music wire, beryllium-copper. No chemical compatibility problems were found except with galvanized wires. Various surface treatments, including coatings of various metals and use of fluxes, were evaluated to determine the combination of wire and surface treatment which would give good wetting and adherence. The copper and beryllium-copper wires proved to give the best wetting and adherence with the least surface treatment. Single-fiber bond strength tests were carried out by casting single filaments into the matrix at various depths. Copper and beryllium-copper wires gave the best results and coating with Cerrobend improved the shear strength considerably. Beryllium-copper wire was chosen because of the high strength, good wetting, and high bond strength with the matrix.

Chopped glass filaments were eliminated because of their poor wetting and bonding characteristics.

Boron filaments showed good single filament shear strengths with the matrix, probably due to the rough surface of the filament; however, wetting was poor and could not be improved by surface treatment. Coating of the boron filament with copper promoted wetting and adherence; however, a reliable process could not be worked out in the time available to coat adequate quantities of the chopped filaments.

Sapphire and silicon carbide whiskers proved to be satisfactory when coated with copper.

The use of oxide microparticles such as aluminum oxide to improve creep resistance and to control grain growth, size, and structure was abandoned because problems which were encountered when attempting to disperse the fine powders into the metal matrix could not be solved in time.

Metallic microparticles of tungsten and boron carbide were acceptable provided they were coated with bright copper.

Final Selection of Sample Materials

The research performed inhouse and by the contractors was reviewed and a final selection of the sample materials made in late November 1970.

The composite materials samples selected for the Apollo 14 inflight demonstration were divided into four general classifications:

a. Precompressed metal powder compacts with dispersed non-melting particles.

b. Matrix materials in combination with various non-melting particles, fibers, whiskers and/or gas.

c. Dispersions of immiscible materials (both melting).

d. Solidification and casting demonstrations.

Because of lack of time during the flight, none of the samples in the group D was processed. Only 11 of the 18 flight samples were processed. A list of these 11 samples is shown in Table 1. A more detailed description follows.

TABLE 1. SAMPLE COMPOSITION

Sample Group	Specimen No.	Contents by Volume	Processing
A. Precompressed Powder Compacts Containing Dispersed Particles	1	70 percent In–Bi Powder, 30 percent W	1. Melt
	2	70 percent In–Bi Powder, 30 percent B_4C	2. Solidify
B. Matrix Material with Fibers, Particles, Whiskers, and/or Gas	4	75 percent In–Bi w/SiC, 25 percent Ar	1. Melt
	5	75 percent In–Bi w/BeCu, 25 percent Void	2. Disperse by Shaking
	7	75 percent In–Bi, 25 percent Ar	3. Solidify
	8	75 percent In–Bi w/W, 25 percent Void	
	10	70 percent In–Bi, 30 percent W	
	11	75 percent Paraffin w/BeCu, 25 percent Void	
C. Immiscible Materials Dispersions	6	50 percent Paraffin, 50 percent NaAc	1. Melt
	9	40 percent Paraffin, 40 percent NaAc, 20 percent Ar	2. Disperse by Shaking
	12	40 percent Paraffin, 40 percent NaAc, 20 percent W	3. Solidify

MATRIX MATERIALS

In–Bi – Indium Bismuth Eutectic Alloy, M. P. 345.15° K (72°C), density 8.0 g/cc

Paraffin – Melting Range 327.15 – 331.15°K (54–58°C), density 0.85 g/cc

NaAc – Sodium Acetate trihydrate, M.P. 331.15°K (58°C), density 1.44 g/cc

DISPERSANTS

W — Copper coated Tungsten microspheres, density 19.3 g/cc

B_4C — Copper Coated Boron Carbide microspheres, density 2.52 g/cc

SiC — Copper Coated Silicon Carbide Whiskers, density 3.6 g/cc

BeCu — Beryllium Copper fibers, density 8.26 g/cc

Ar — Argon gas

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Preparation of Samples

A ground control, flight, and spare for each of the samples was prepared. All were prepared at MSFC with the exception of the powder compacts. The procedures followed in preparing the 11 samples are given below.

Precompressed Powder Compacts. The powder compacts for these samples were prepared by Cornell Aeronautical Laboratory. The detailed procedures for preparing these samples is given in Reference 4. A brief description of these procedures follows.

Sample 1: 70 Percent In-Bi Powder, 30 Percent Tungsten Particles (by Volume). A mixture of 30 vol percent spherical copper-coated tungsten particles and 70 vol percent In-Bi eutectic powders was compressed to form a powder compact. The In-Bi powders and tungsten particles were of the same size, -325 mesh. The In-Bi powders were leached in a 3-percent solution of hydrochloric acid to remove oxides. The copper-coated tungsten particles were heated in a hydrogen atmosphere to reduce oxides. The powders were weighed out, placed in a sealed container, and mixed for 4 hr to obtain a random distribution. The mixed powders were then compressed in a double acting die to form a powder compact 1.74 cm in diameter and 7.6 cm long with a theoretical density of 96 percent.

Sample 2: 70 Percent In-Bi Powder, 30 Percent Boron Carbide Particles (by Volume). A mixture of 30 vol percent spherical copper-coated boron carbide (B_4C) particles and 70 vol percent In-Bi eutectic powders was compressed to form a powder compact. The B_4C particles and In-Bi powders were of the same size, -325 mesh. The particles and powders were processed in the same manner as the contents of Sample 1. The mixed powders were compressed to form a compact 1.74 cm in diameter and 2.5 cm long with a theoretical density of 89 percent. In trying to compress compact to this lower density, it was impossible to make one long enough to fill the capsule. Therefore, three compacts of the stated size were used in each capsule.

Note: Each sample was inserted into standard aluminum capsule which was then sealed by electron beam welding.

Matrix Materials with Fibers, Particles, Whiskers and/or Gas

Sample 4: 71 Percent In-Bi, 4 Percent SiC Whiskers, 25 Percent Argon (by Volume). Approximately 1.5 g of copper-coated SiC whiskers (1-10 μ diam. × 20-400 μ long) and 90 g of liquid In-Bi were premixed, poured into a heated aluminum capsule, and allowed to solidify. The actual whisker content amounted to approximately 4.0 percent by volume.

Sample 5: 68 Percent In-Bi, 7 Percent BeCu Fibers, 25 Percent Argon (by Volume). The sample was prepared by placing approximately 7 g of Cerrobend coated beryllium-copper fibers (.13 mm diam. × 2.5 mm long) into a heated aluminum capsule and then pouring in approximately 97 g of In-Bi eutectic. The actual fiber content amounted to approximately 6.6 percent by volume.

Sample 7: 75 Percent In-Bi, 25 Percent Argon (by Volume). The samples consisted of 75 vol percent In-Bi and 25 vol percent argon gas. The sample was prepared by pouring approximately 100 g of molten In-Bi into a preheated capsule and inserting a preheated stainless steel mixing screen while the In-Bi was molten.

Sample 8: 67 Percent In-Bi, 8 Percent Tungsten Particles, 25 Percent Argon (by Volume). The sample was prepared by pouring approximately 21 g of copper-coated spherical tungsten particles (approximately 100 μ diam.) into a preheated aluminum capsule and then adding approximately 99 g of molten In-Bi eutectic. The actual particle content amounted to approximately 8.2 percent by volume.

Sample 10: 70 Percent In-Bi, 30 Percent Tungsten Particles (by Volume). Approximately 105 g of copper-coated spherical tungsten particles (100 μ diam.) were placed in a preheated capsule, a tungsten mixing pellet added, and then 100 g of In-Bi eutectic poured into the capsule. The actual particle content amounted to approximately 30.4 percent by volume.

Sample 11: 68 Percent Paraffin, 7 Percent BeCu Fibers, 25 Percent Argon (by Volume). Approximately 7 g of Cerrobend-coated BeCu fibers were placed in a preheated capsule, and 10 g of paraffin were then poured into the capsule. The actual fiber content was approximately 6.7 percent by volume.

Note: After cooling and solidification of the sample, each capsule was evacuated, backfilled with argon, if required, and sealed by electron beam welding.

<u>Immiscible Materials</u>

Sample 6: 50 Percent Paraffin, 50 Percent Sodium Acetate (by Volume).

Sample 9: 40 Percent Paraffin, 40 Percent Sodium Acetate, 20 Percent Argon (by Volume).

Sample 12: 40 Percent Paraffin, 40 Percent Acetate, 20 Percent Tungsten Spheres (by Volume).

Preparation was similar in all cases except for the addition of argon in Sample 9 and the 100-μm spherical tungsten particles in Sample 12. The procedures used were as follows:

The sodium acetate trihydrate was prepared by melting at 341.15° K (154.4° F) and then filtering to remove any unmelted crystals. The melted, filtered sodium acetate trihydrate was then poured into a preheated capsule to the desired level and the tungsten mixing pellet added. In Sample 12, the tungsten particles were added at this point. Then melted paraffin was added to the desired level and allowed to solidify. A needle was used to puncture the paraffin to admit air for equalization of pressure to insure solidification of the sodium acetate. It was discovered that sodium acetate has a tendency to supercool at lowered vapor pressures caused by the solidification shrinkage in a sealed container. This phenomenon deserves further explanation, which should be given before future use of this material as a model matrix is recommended. At this point Sample 9 was backfilled with argon.

Note: All capsules were sealed by electron beam welding.

SAMPLE EVALUATION AND RESULTS

The evaluation plans provided that one half of each sample, flight and ground processed control, would be delivered to a contractor investigator and the other half retained at MSFC for evaluation. Contractor participation in the evaluation was as follows:

Cornell Aeronautical Laboratory	Samples 1, 2, and 10 [4]
Arthur D. Little, Inc.	Samples 4, 7, and 8 [2]
General Dynamics–Convair	Samples 5 and 11 [3]
TRW Systems	Samples 6, 9, and 12 [5]

Preliminary evaluation of all samples was conducted at MSFC with the contractors present during evaluation of the samples assigned to them. Preliminary evaluation included:

a. Radiography of the unopened capsules to show the distribution of sample materials within the capsule.

b. Opening of the capsules.

c. Documentation (photography, weighing, measuring, etc.) and identification of the sample.

d. Longitudinal halving of the samples.

e. Additional photographs of the halved samples.

Additionally for those samples containing argon gas, the capsules were accurately weighed to 0.1 mg before opening; the capsules were then pierced and reweighed. By comparing weights before and after piercing, it was determined whether the gas had been retained during the welding process.

A summary of the results of the detailed evaluation of the samples follows:

Precompressed Powder Compacts Containing Dispersed Particles

The primary objective for processing this group of samples was to observe the redistribution of particles in a powder metal matrix melted and solidified in a weightless environment. The density of the non-melting particles differed significantly from that of the matrix (approx. 1/2.5). Significant segregation of particles had been expected when the samples were melted under gravity.

Processing of specimens in this group was accomplished by placing the sample capsule in the heater, heating for 10 min to melt the In-Bi matrix, and cooling on the heat sink for 30 min. The ground control samples were heated in the horizontal position and cooled in the vertical position. A temperature profile, Figure 5, taken during processing of ground control Sample 1 shows that the matrix was molten after about 6 min and was solidified after about 20 min. None of these samples was shaken.
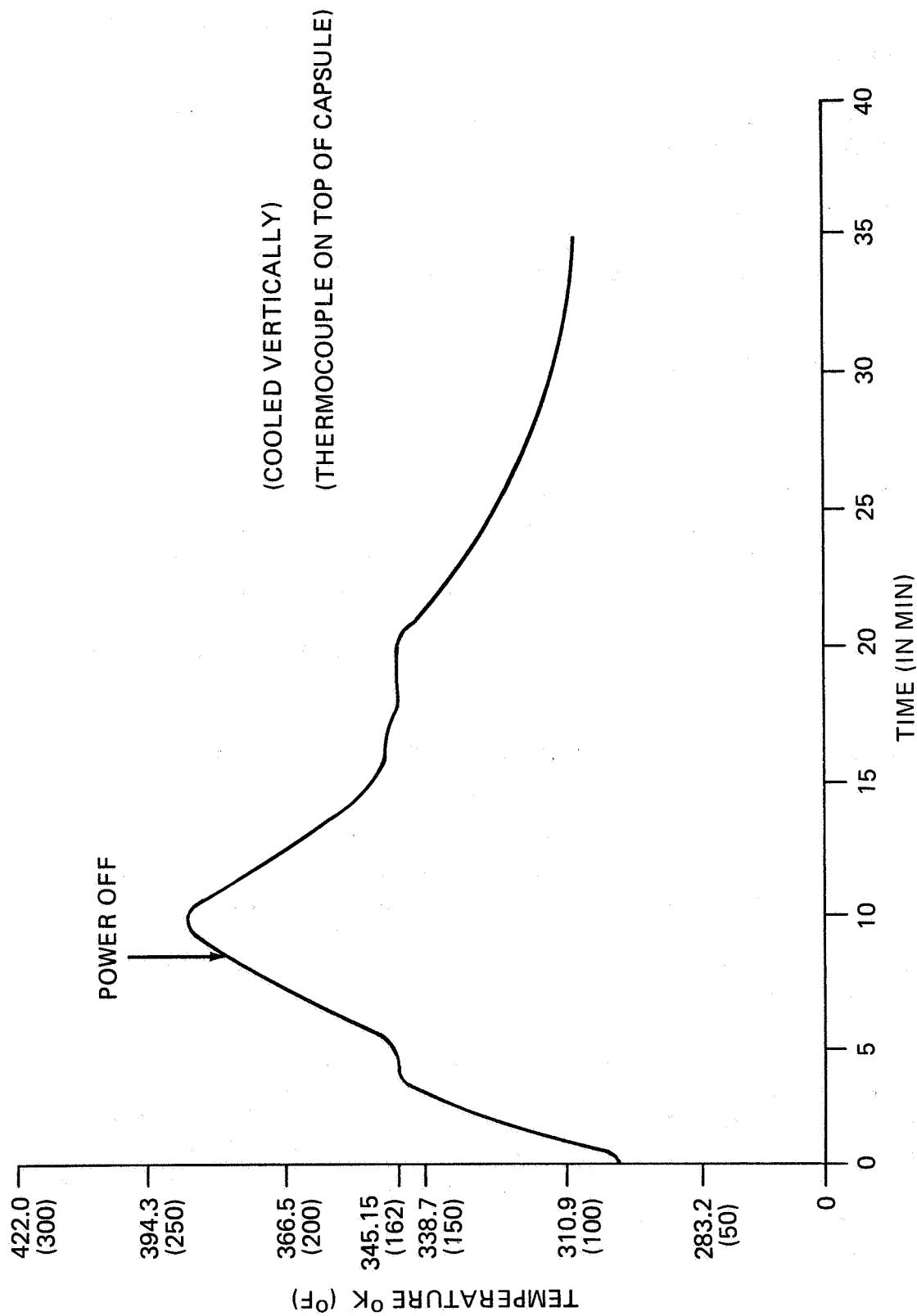
Figure 5. Heat test on Specimen 1C in qual heater.

20

A comprehensive report on characterization of the samples in this group is given in Reference 4. Only a summary of those studies and results is given here.

Sample 1: 70 Percent In-Bi, 30 Percent Tungsten Powder Compact. Tungsten particle distributions were investigated in three sample half sections identified as:

Developmental Sample, 1D-A-00, in the as-pressed condition.

Control Sample, 1C-A-00, melted and solidified under gravity.

Flight Sample, 1F-A-00, melted and solidified under near zero g.

Procedure for Determining Tungsten Particle Distribution. The distribution of tungsten particles was determined by the following procedure:

a. The exposed planes were mechanically polished to produce a surface of particles and matrix.

b. Photomicrographs (100X) of the prepared surfaces were taken at intervals in both the directional (longitudinal) and cross-directional (radial) directions. Figure 6 shows the typical method used in documenting the particle distributions.

c. The 100X photomicrographs were enlarged 2.5 times to produce large negatives [22.23 × 28.58 cm (8.75 × 11.25 in.)]. Particle distribution was determined by superimposing a 1-cm-ruled grid on vellum over the negative and marking with a pencil each grid point occupied by a particle. The total number of occupied points divided by the total number of points gave the volume percent of particles for the particular area.

d. The data was then plotted to show particle distribution in both directional (longitudinal) and cross directional (transverse) directions for each sample.

Tungsten Particle Distribution. The directional display of tungsten particle distribution in the three samples is shown in Figure 7. Because of the method of compaction, the tungsten particle distribution in the "as-pressed" sample was not uniform. There was a noticeable decrease in particle density at the ends of the developmental sample (Fig. 8). In the cross-directional (transverse) direction distribution shows the particle content to be consistently lower at the edges than at the center. Data taken along the center axis compared very well with the directional data.
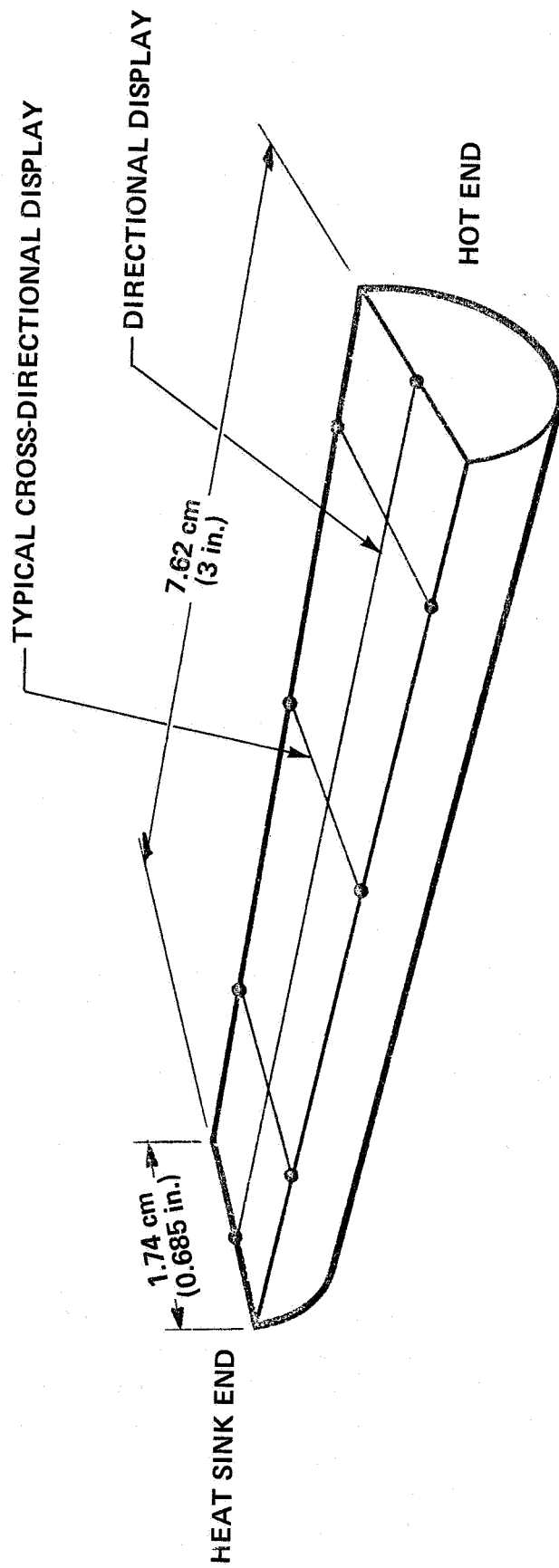
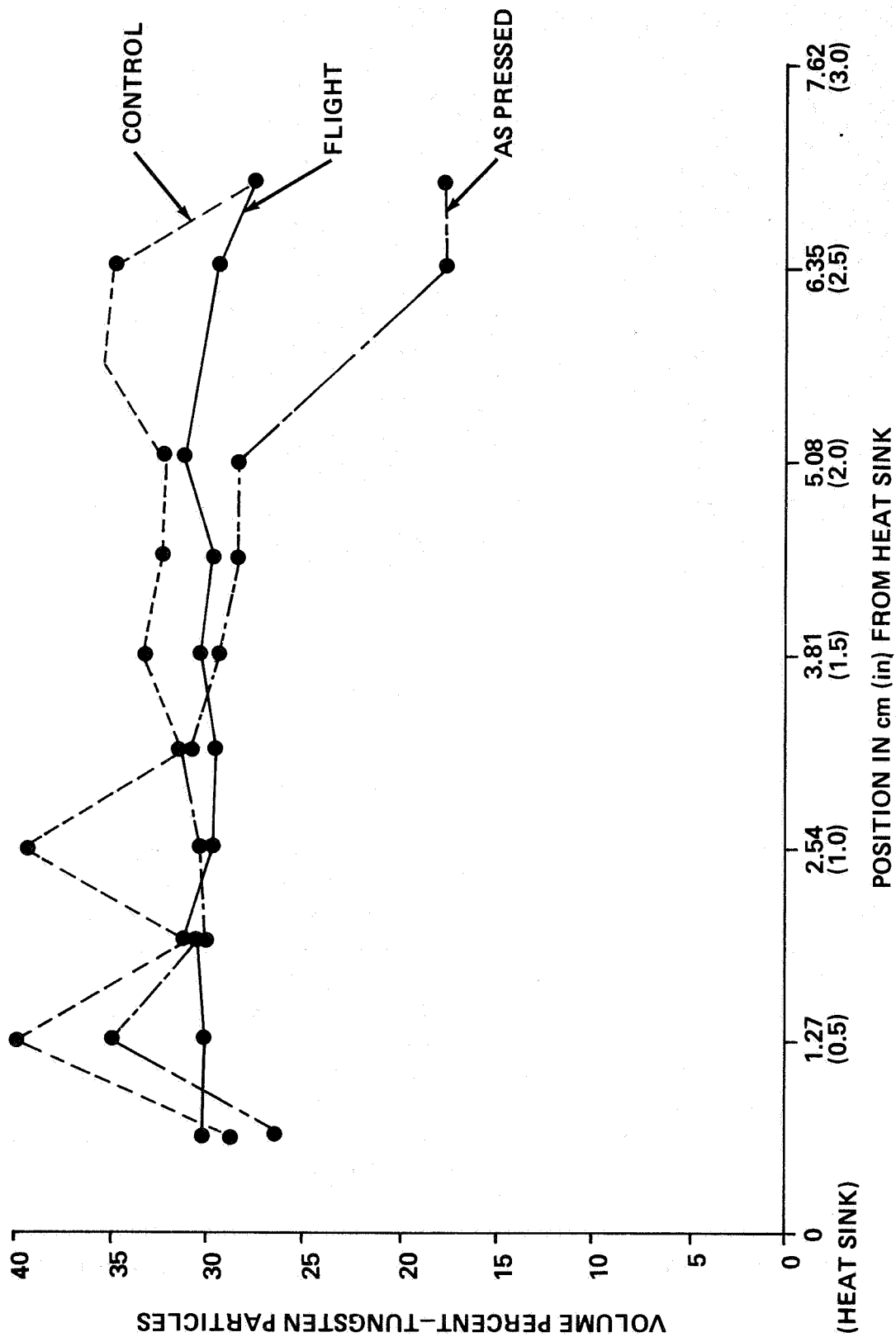Figure 6. Schematic of photographic documentation.

Figure 7. Sample 1 – directional display observed (data from Cornell Aeronautical Laboratory).
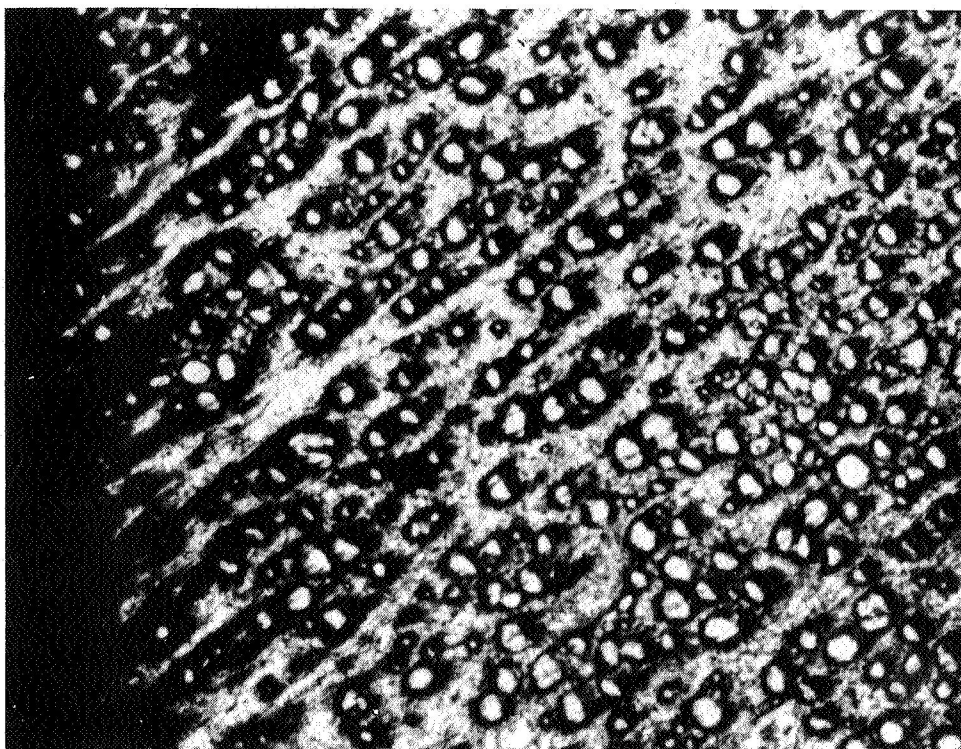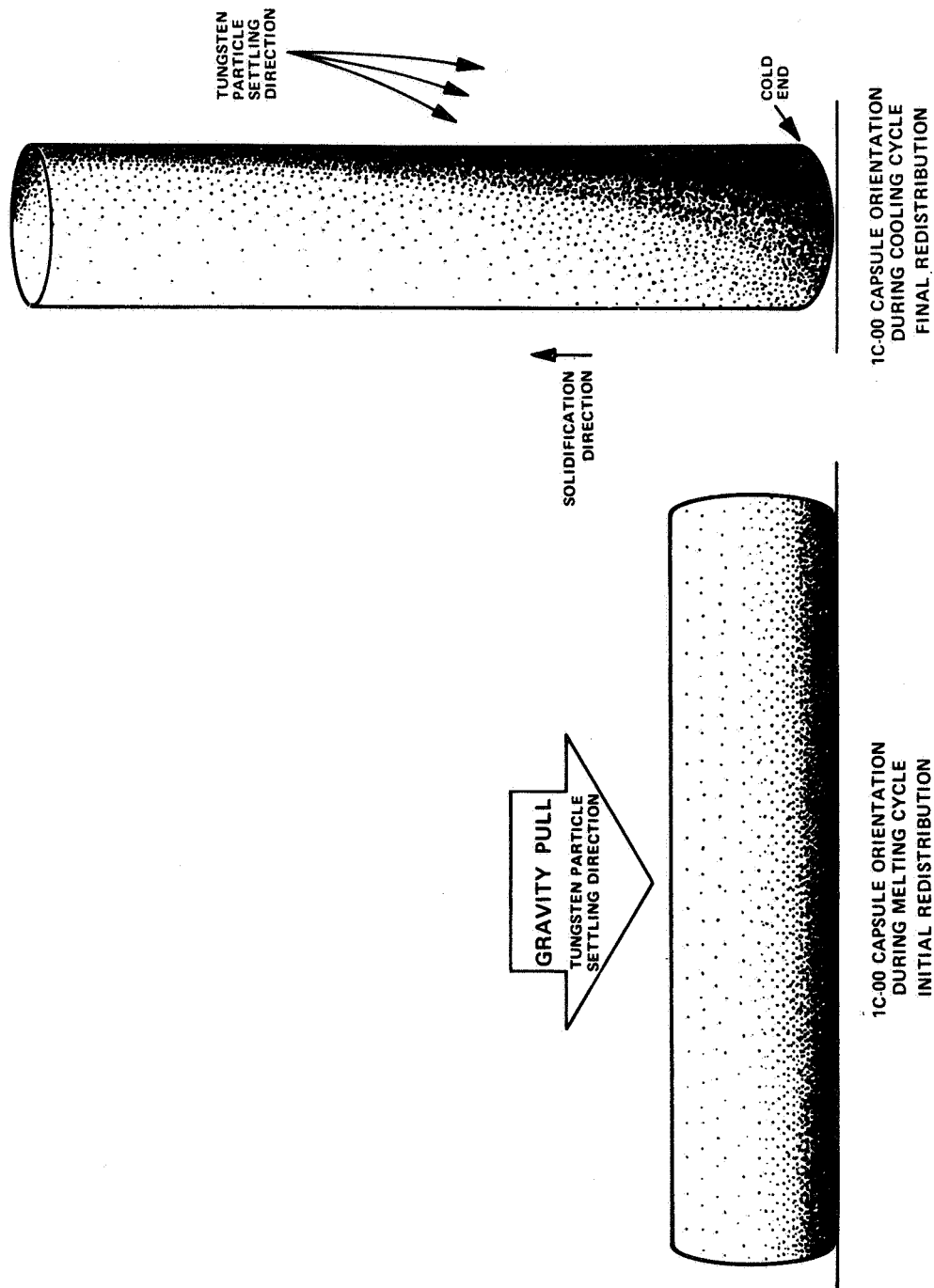
Figure 8. Developmental Sample 1 (1D-A-00) directional display,
photomicrograph, (100X).

In the control sample there was an increase in particle density toward the center of the sample and toward the heat sink (gravity direction). Abrupt and large changes in volume percent tungsten indicate that some particle settling started but complete segregation did not occur. It was thought that this was due to the limited time above the melting temperature while in the vertical position. However, subsequent tests performed at MSFC (Appendix) have shown that complete segregation does not occur after prolonged heating in the vertical position. The mechanisms which prevent segregation are not understood. The method of processing the ground control sample may have further disturbed the distribution. It was melted in the horizontal position and then moved to the vertical position for cooling. Therefore a settling pattern as shown in Figure 9 may have occurred. The orientation of the sample during heating with respect to the cutting plane is not known.

In the flight sample, a more homogeneous distribution of particles was seen in the longitudinal display. This display was the result of an actual count

TUNGSTEN
PARTICLE
SETTLING
DIRECTION

COLD
END

GRAVITY PULL
TUNGSTEN PARTICLE
SETTLING DIRECTION

SOLIDIFICATION
DIRECTION

1C-00 CAPSULE ORIENTATION
DURING MELTING CYCLE
INITIAL REDISTRIBUTION

1C-00 CAPSULE ORIENTATION
DURING COOLING CYCLE
FINAL REDISTRIBUTION

NOTE: TUNGSTEN PARTICLES REPRESENTED BY BLACK STIPPLING

Figure 9. Probable tungsten particle redistribution during processing
of control sample (1C-00).

taken at 15 different locations along the axis. As seen in Figure 7, the concentration of tungsten particles was very nearly constant at 30 vol percent which was the initial concentration. Good correlation was obtained between the cross directional data at the intersection of the center axis and that taken in the directional display along the axis.

Surface Features. In comparing the surfaces of the samples in contact with the capsule wall, some interesting differences were noted between the appearance of the control sample and the flight sample. Surface distortions or depressed regions were seen in the flight samples and not in the control samples (Figs. 10 and 11). It was postulated that these depressed regions were the result of bubbles or voids moving to the surface of the sample and the interaction between the tungsten particles and associated void volumes.

Sample 2: 70 Percent In-Bi, 30 Percent B_4C Powder Compact. Three samples were again evaluated:

Developmental Sample, 2D-A-00, as pressed.

Control Sample, 2C-A-00, melted and solidified under gravity.

Flight Sample, 2F-A-00, melted and solidified under near zero g.

These samples were evaluated in the same manner as Sample 1. The control and flight samples consist of three individual segments. Upon removal from the capusles, the three segments remained separate indicating that complete melting had not occurred. Some melting was evident in the segment at the bottom or heat sink end of the samples in that some metal had flowed from the compact. More metal had flowed from the flight sample than from the control sample. The distribution of the B_4C particles in two segments of the flight samples was different from the developmental sample indicating that some melting had occurred (Fig. 12).

Summary of Group A Results. The distribution of tungsten particles in Sample 1 was determined quantitatively using a point-intercept method. The results obtained indicate the following:

a. When compared with the development sample, it was found that a redistribution of tungsten particles had occurred after melting in both the control and flight samples.

26
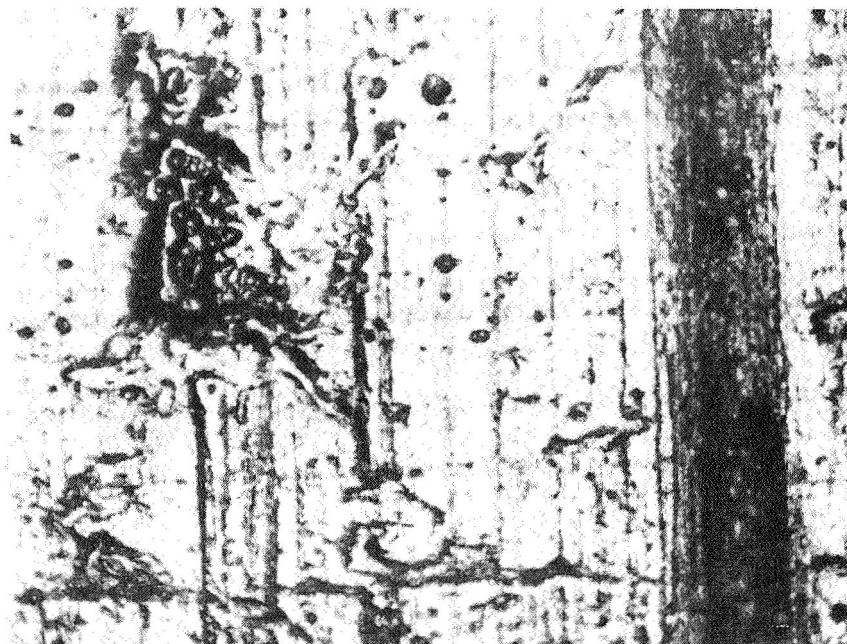
4
(A)
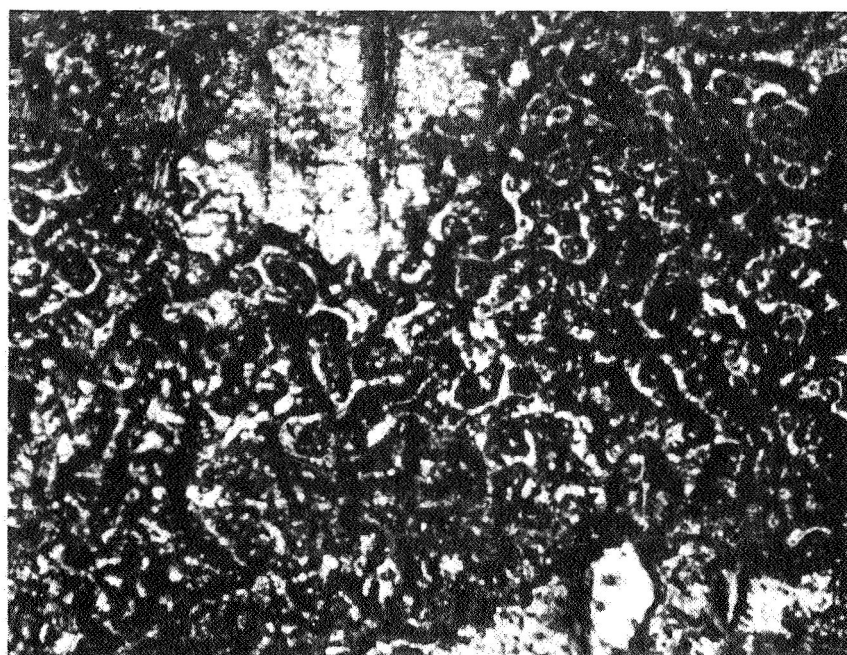
4
(B)

Figure 10.   Flight Sample 1 (1F-A-00) surface
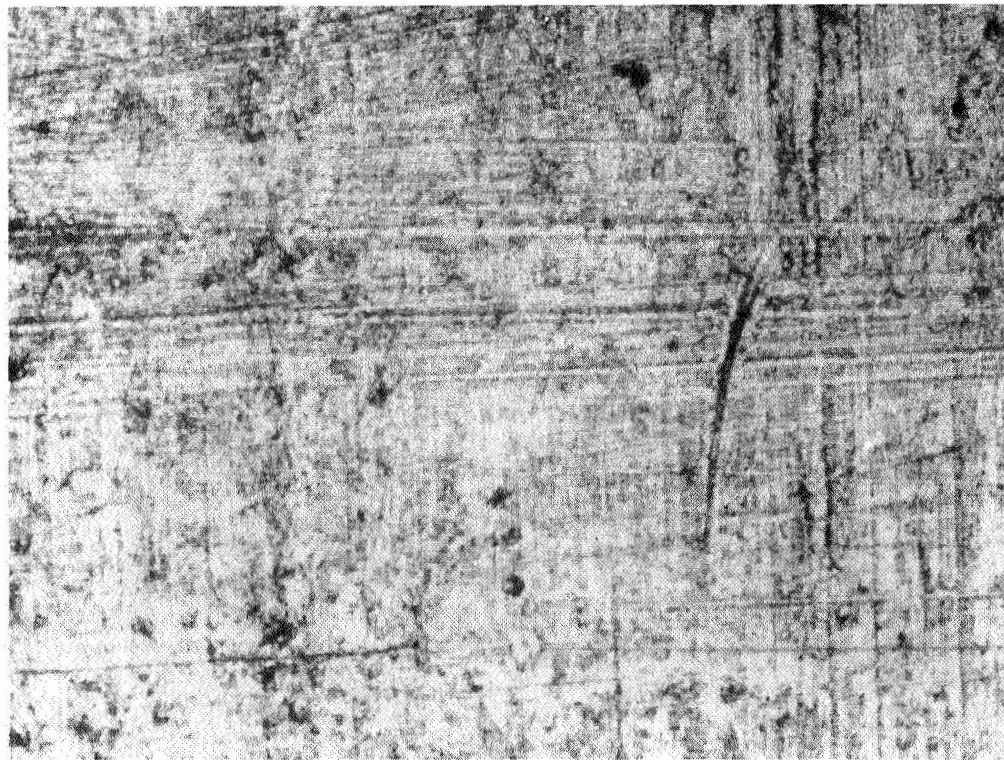distortion, photomicrographs, (100X).

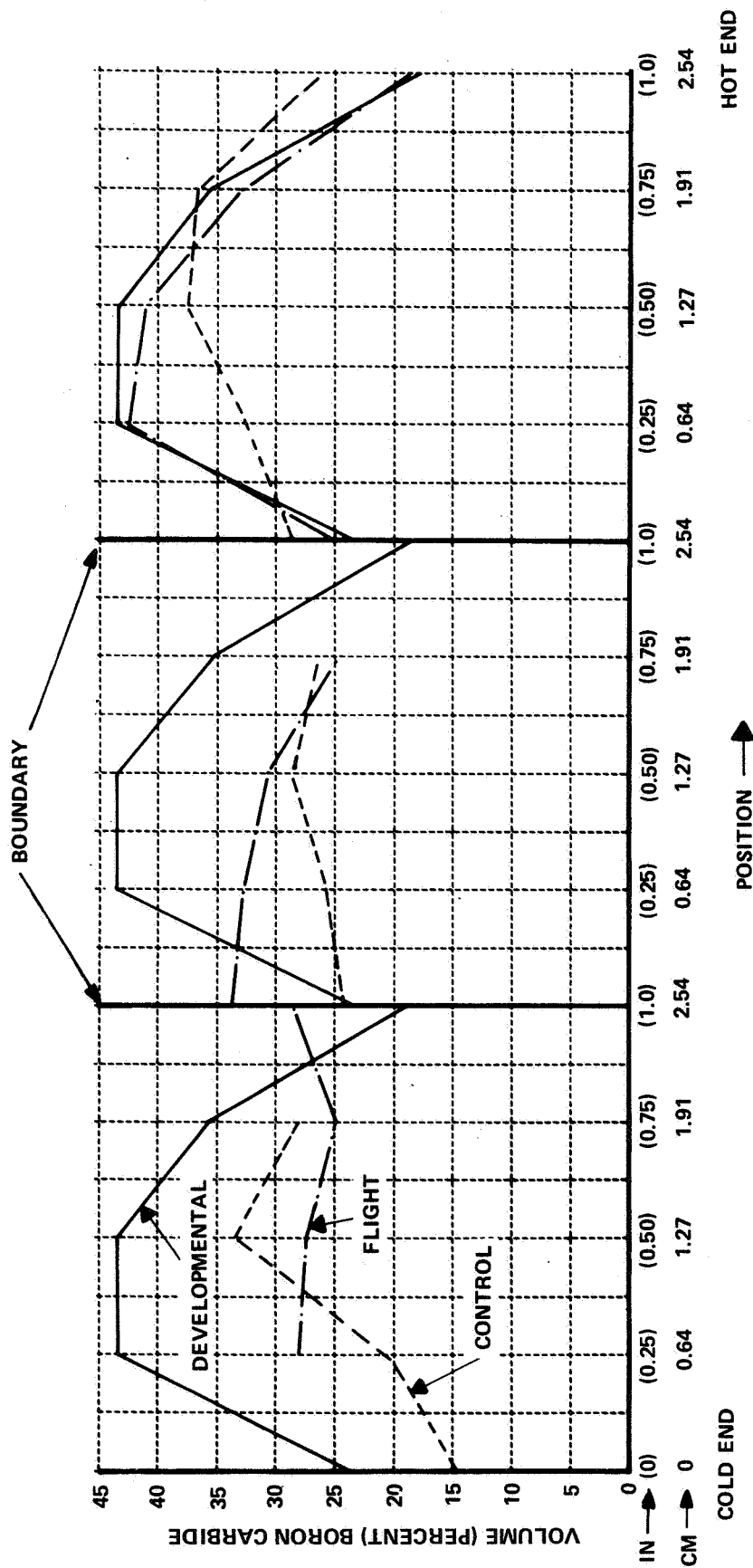Figure 11. Control Sample 1 (1C-A-00) surface at center (100X).

Figure 12.   Sample 2 – specimen position in capsule.

b. The distribution obtained in flight Sample 1 was more uniform than that obtained in control Sample 1. This indicates that there are advantages to be gained in liquid-phase sintering in the absence of gravity forces. This also has implications for the composite casting processes.

The distinct differences in surface features of the control and flight sample indicate differences in bubble/void motion and interaction between bubbles/voids and particles in zero g compared with gravity conditions. Additional experiments may be warranted to determine what mechanisms are at work and the implications for space manufacturing.

The quantitative data on Sample 2 are obscured by the evidence of incomplete melting; however, it seems that in those segments which had partially melted, the distribution of B_4C particles tended towards greater uniformity.

Matrix Material with Fibers, Particles, Whiskers and/or Gas.

The primary objectives in processing this group of samples was to demonstrate the dispersion of high and low-density solid reinforcements and/ or gases in a liquid phase matrix and the stability of such mixtures during solidification in a near weightless environment.

Processing of the flight and ground control samples in this group was accomplished by placing the capsule containing the sample in the heater, heating for 10 min to melt the matrix, shaking by hand to disperse the solids and gases in the liquid, and cooling on the heat sink for 30 min.

Sample 4: 71 Percent In-Bi, 4 Percent SiC Whiskers, 25 Percent Argon. The objective was to achieve a stable dispersion of nonmetallic whiskers and gas bubbles in a metal matrix through processing in a weightless environment.

Radiographs of the unopened control and flight samples are shown in Figure 13. The down direction for the control sample, and the heat sink position in both cases, is at the bottom of the picture. The solid is in contact with the capsule walls and bottom in the control sample. The flight sample is in contact with both ends of the capsule. There is a minor neck near the heat sink end and a major neck at the opposite end where there is no contact with the capsule walls. This was verified upon removal of the sample from the capsule (Fig. 14). Both ends of the flight sample and one end of the control sample were shiny, indicating contact with the capsule.
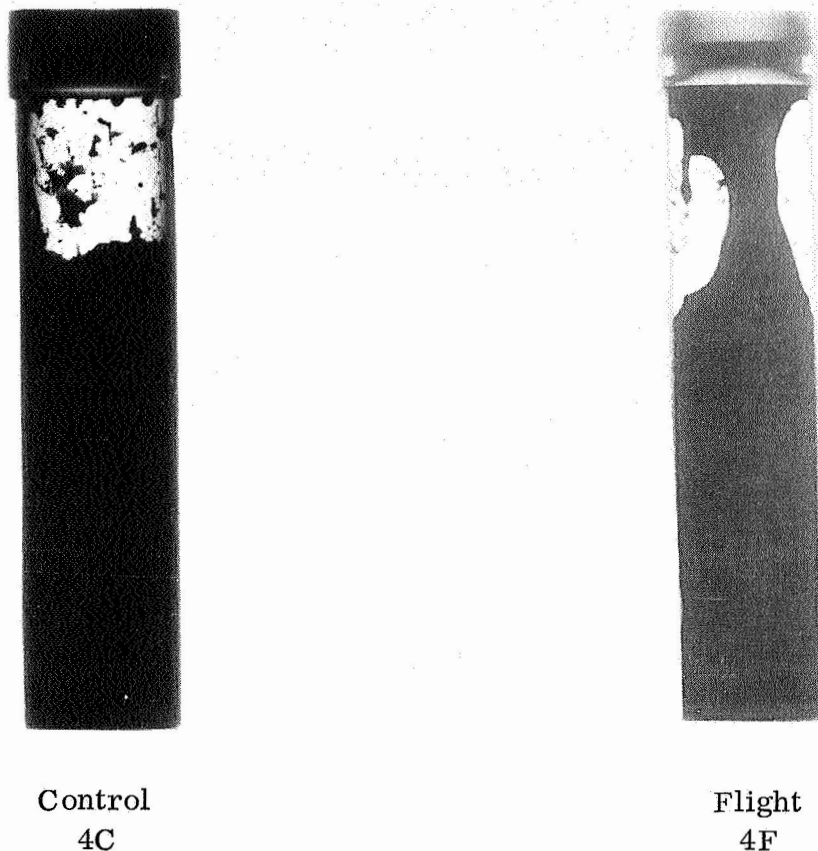
Control   Flight
4C   4F

Figure 13. X-ray radiographs, Samples 4C and 4F.

The sample was sectioned longitudinally and metallographically prepared. The most significant difference between the flight and control samples is seen in Figure 15. Approximately the bottom fifth of the control sample was fully dense with no SiC whiskers or argon bubbles. In contrast, the flight sample was uniform throughout, both in microstructure and in distribution of gas pores.

Pore density and pore-size distribution measurements were performed to evaluate differences between the flight and control samples. The flight sample had a pore density approximately twice that of the control sample. Furthermore, the pore density of the flight sample was rather uniform, varying from 16 percent near the bottom to 19 percent at the top. In contrast, the control sample varies from 13 percent near the bottom of the pore containing region to 5 percent at the top.
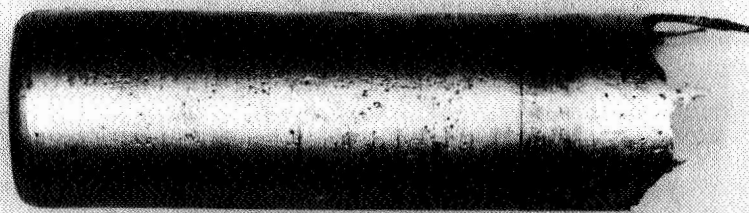
Specimen No. 4C-00	Sample Configuration
as filled	X Complete
X ground processed	Segment
flight processed	Section
View: top bottom	
Side (0)/90/180/270°	
Enlargement: x	
cm 2 3 4 5	6 7 8 9 10
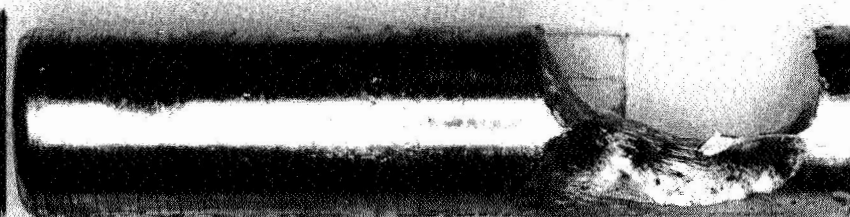
Specimen No. 4 F-00	Sample Configuration
as filled	X Complete
ground processed	Segment
X flight processed	Section
View: top bottom	
Side 0/90/(180)/270°	
Enlargement: x	
cm 2 3 4 5	6 7 8 9 10

Figure 14. Side views of Samples 4F-00 and 4C-00 after removal from capsule.

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Control   Flight
4C   4F
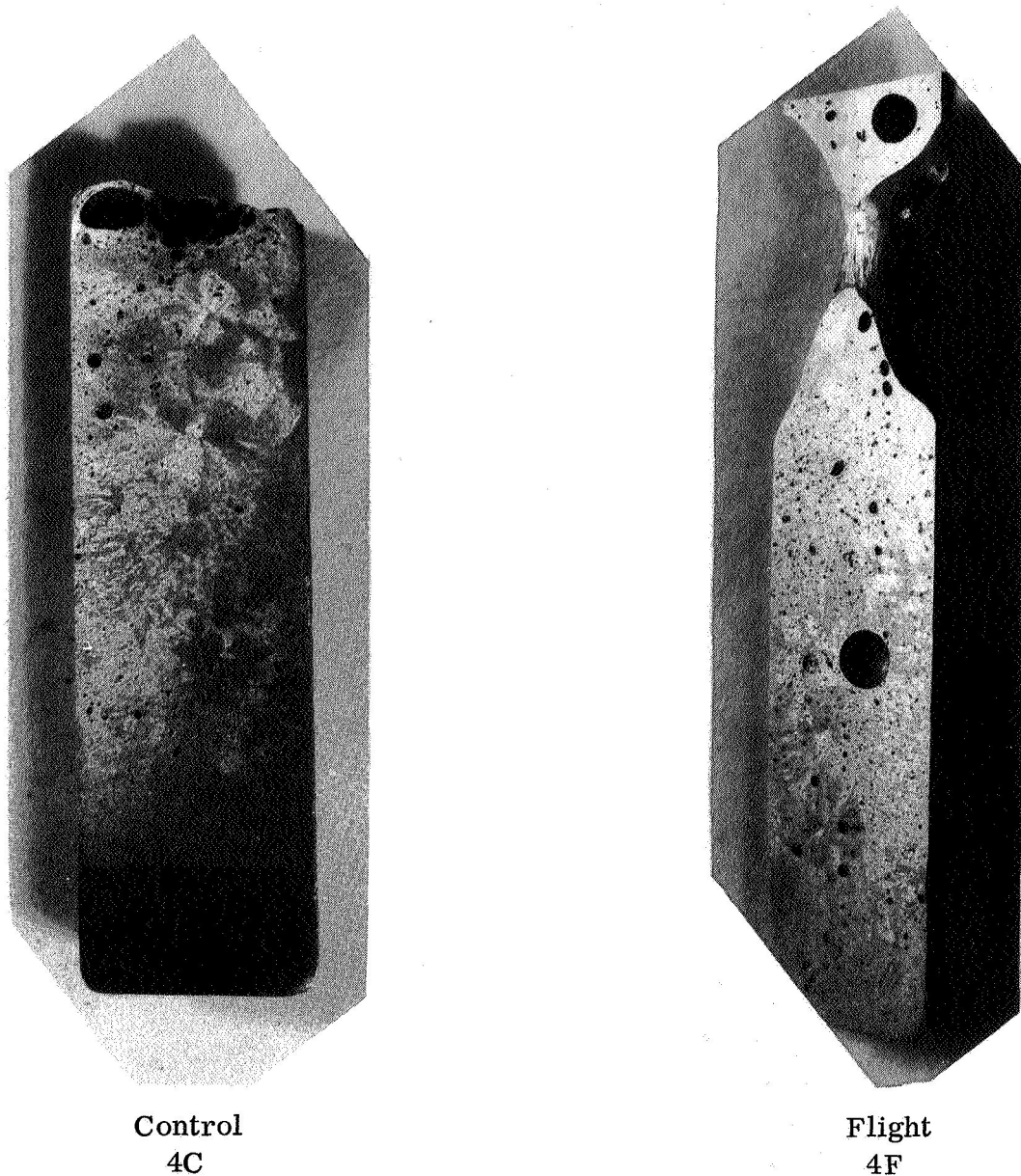
Figure 15.   Photomacrographs of longitudinal sections,
Samples 4F and 4C, etched ~ 1.5X, (ADL Photos).

The whiskers did not appear to be wetted by the molten alloy, since most ended up on the outside of the sample, on the gas pore surfaces, or on the shrinkage pore surfaces.   There was no alignment of the whiskers with the eutectic lamellae.

<u>Sample 5: 68 Percent In-Bi, 7 Percent BeCu Fibers, 25 Percent Argon.</u>
The objective was to achieve a stable dispersion of metallic fibers and gas
bubbles in a metal matrix through processing in a weightless environment.

It was determined that the argon gas had leaked from these samples
during electron beam welding of the capsules.

Since the sample occupied only 75 percent of the capsule volume, it
was allowed to move freely within the capsule. Radiographs of the unopened
capsules (Fig. 16) show that the control sample, as was to be expected,
settled to the bottom. The flight sample was free floating upon solidification,
with contact only at the walls and slightly at one end. Note the spherical
shape of the free end of the flight sample. Examination of the surfaces of the
samples after opening shows that the flight sample was not in complete contact
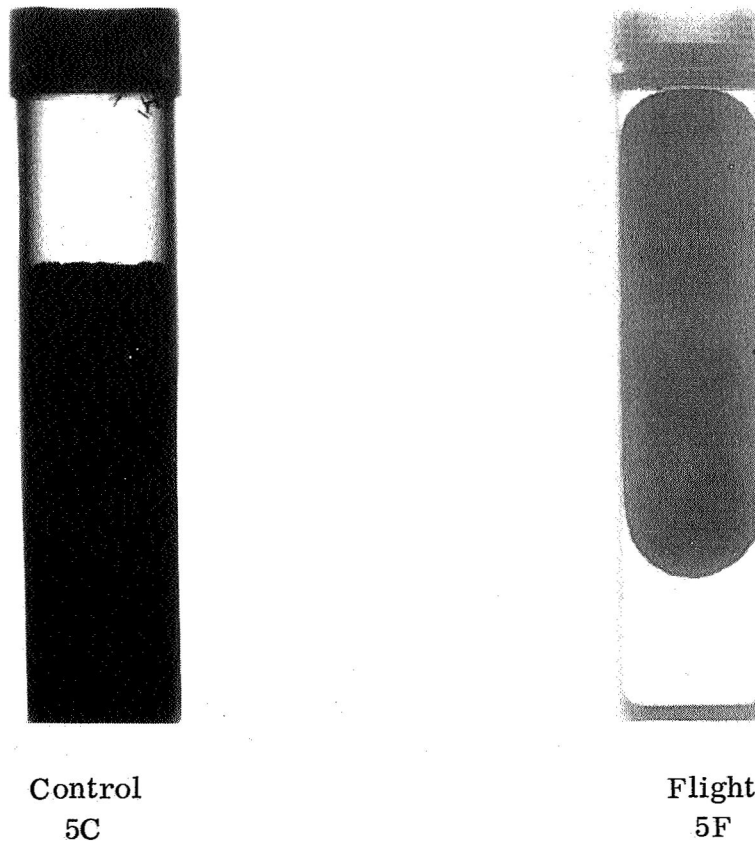with the walls of the capsule (Fig. 17).

Control   Flight
5C   5F

Figure 16. X-ray radiographs, Samples 5C and 5F.

34

The fiber dispersion was quite different in the flight sample, as seen in the cross section in Figure 18, and the neutron radiograph of an .318 cm (0.125 in.) thick section in Figure 19. Gravity induced segregation was evident in the control sample even though the density difference between the fibers and matrix was only 1 percent. The bulk of the fibers settled to the bottom with a small number carried to the top by rising bubbles and surface tension. The fibers were dispersed over the entire cross section of the flight sample. The difference in dispersion is seen in the plot of fiber count over the length of the specimen (Fig. 20).
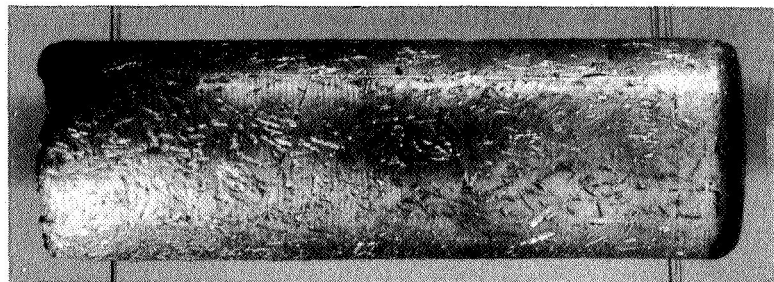
The surface features of the flight sample were quite different from the control sample. There were numerous depressions and pocket formations in the surface of the flight sample, and there was a tendency of fiber alignment, particularly on the free end. Within the flight sample there was some tendency for fibers to coagulate at void interfaces.

Sample 7: 75 Percent In-Bi, 25 Percent Argon. The objective was to achieve a stable dispersion of gas bubbles in a metal matrix through processing in a weightless environment in order to demonstrate to what extent the presence of bubble stabilizing dispersion of particles, fibers, whiskers are to be considered for further process development.
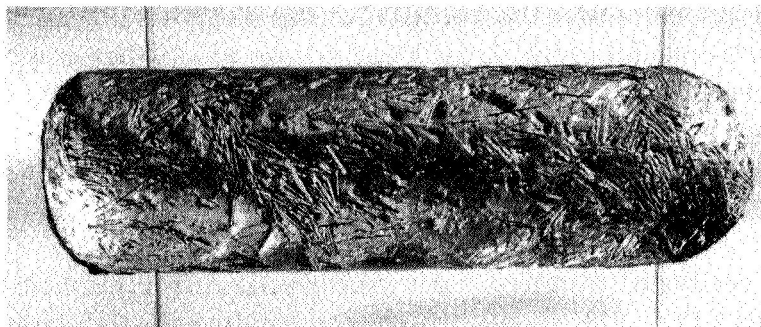
The radiographs (Fig. 21) show the position of the samples within the capsules. The flight sample was touching at isolated points along the capsule walls whereas the control sample had settled to the bottom and was in contact along the walls. The supports for the stainless steel mixing screen are also seen. The differences are further illustrated in the photographs made after removal from the capsules (Fig. 22).

The samples were sectioned longitudinally and prepared for metallography. Photomacrographs (Fig. 23) show the differences in solidification patterns. Solidification in the control sample progressed from the heat sink end, the walls, and the screen. In contrast, solidification in the flight sample was initiated from the different points at which the melt was in contact with the walls. It is believed that this is the cause of the "rosette"-like structure seen in Figure 24.

There was no observable foam or gas porosity in the control sample. In contrast, the flight sample retained approximately 3 percent in the bottom half and 5 percent in the top half. Scanning electron beam photographs of the fractured surface of the flight sample adjacent to the mixing screen (Fig. 25) revealed the presence of many pores.
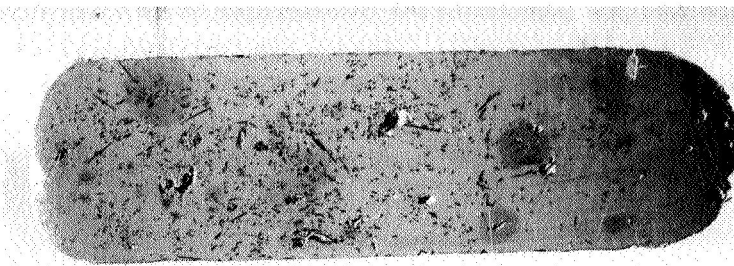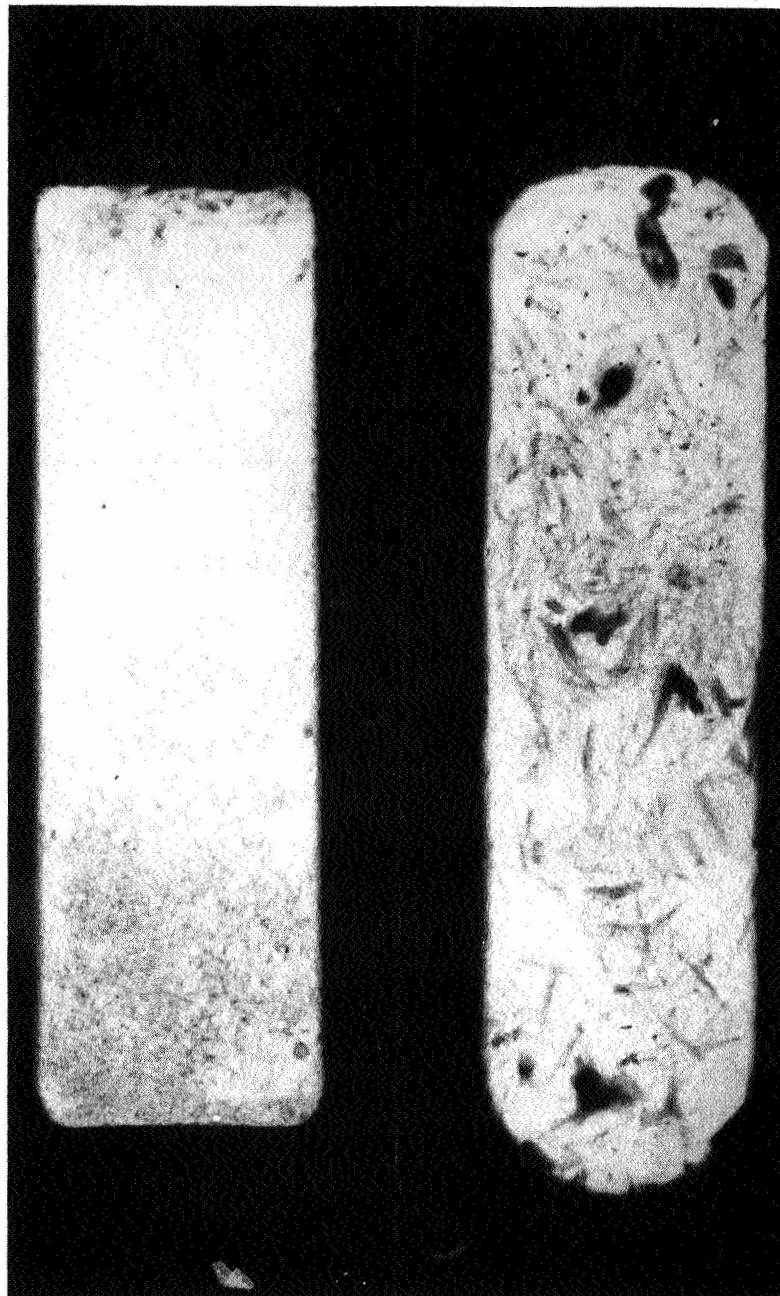
Flight 7F

Control 7C

Figure 18. Photomacrographs of longitudinal sections, Samples 7C and 7F, etched, ~ 1.5X.

Flight 5F

Control 5C (Heat Sink)

Figure 17. Surfaces of Samples 5C and 5F.

36

PLUG END
(TOP)

HEAT SINK END
(BOTTOM)

Control   Flight
5C   5F

Figure 19. Neutron radiograph of .318 cm (1.125 in.)
longitudinal slice, Sample 5.
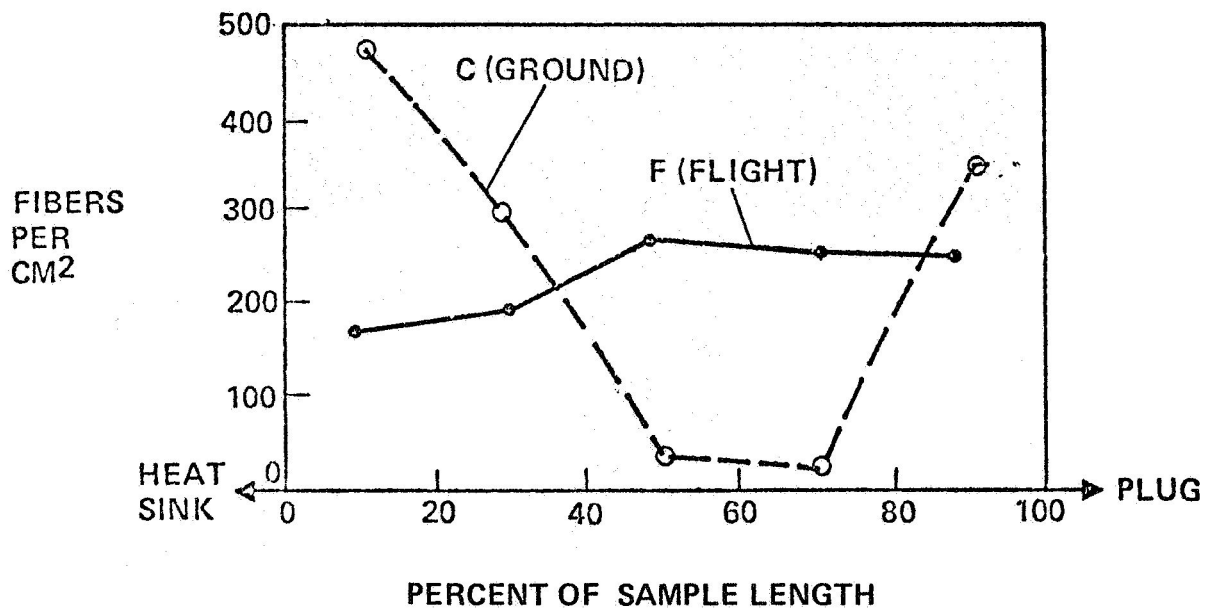
37

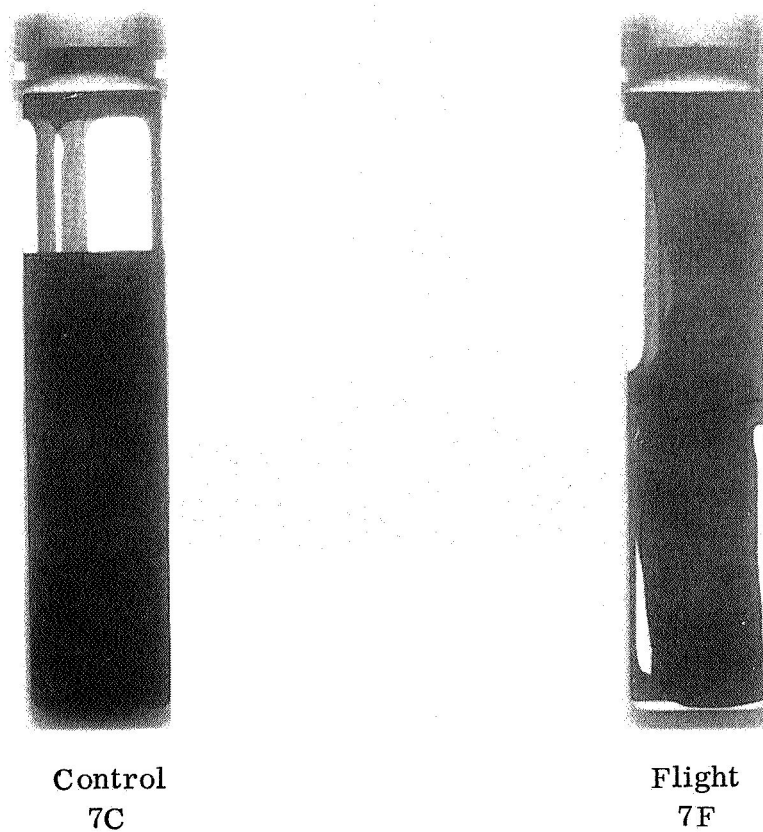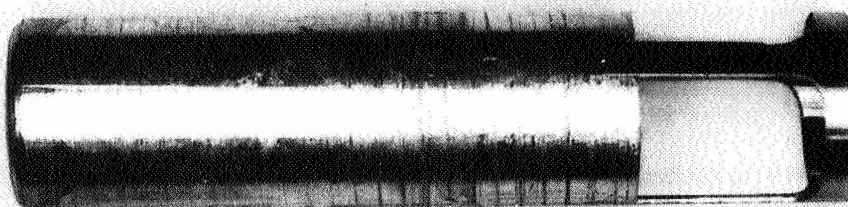Figure 20. Sample 5, fiber dispersion by cross-section count.

Control
7C

Flight
7F

Figure 21. X-ray radiographs, Samples 7C and 7F.

Specimen No. 7C-00	Sample Configuration
as filled	X Complete
X ground processed	Segment
flight processed	Section
View: top bottom	
Side ⓪/90/180/270°	
Enlargement: x	

cm | 2 | 3 | 4 | 5 | 6 | 7 | 8 | 9 | 10

Specimen No. 7F-00	Sample Configuration
as filled	X Complete
ground processed	Segment
X flight processed	Section
View: top bottom	
Side ⓪/90/180/270°	
Enlargement: x	

cm | 2 | 3 | 4 | 5 | 6 | 7 | 8 | 9 | 10

Figure 22. Side views of Samples 7F-00 and 7C-00
after removal from capsules.
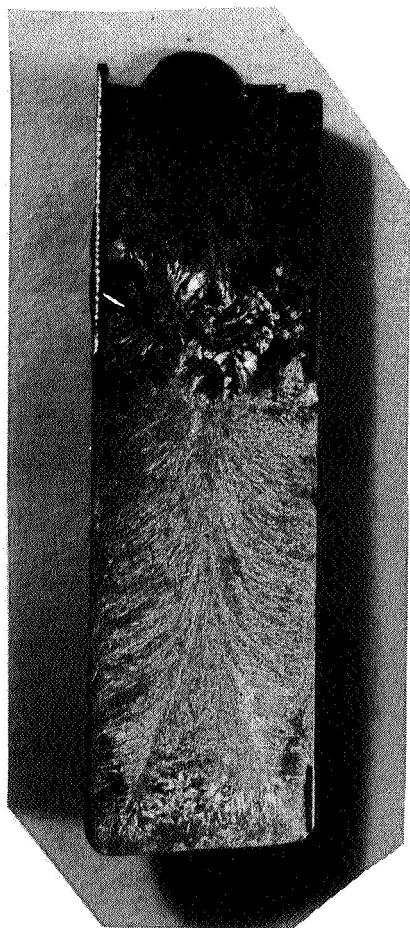
Control
7C
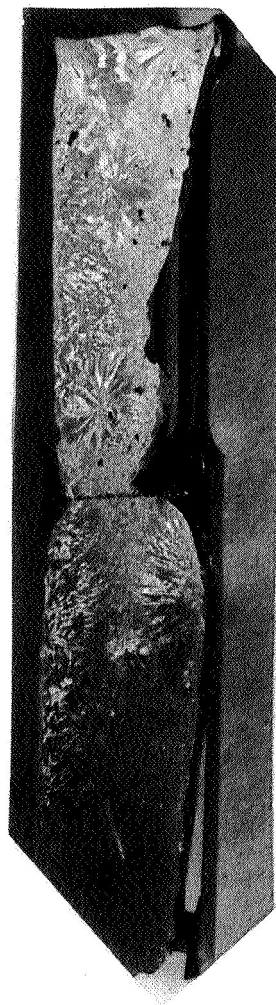
Flight
7F

Figure 23. Photomacrographs of longitudinal sections,
Samples 7C and 7F, etched, ~ 1.5X.

BOTTOM

Figure 24. Flight Specimen 7F-A-00 macrostructure with rosette structures.

41

30X

75X

Figure 25. Scanning electron micrographs of fractured surface
adjacent to stainless steel screen, Sample 7F-A-03.

42

400X

790X

Figure 25 (concluded).

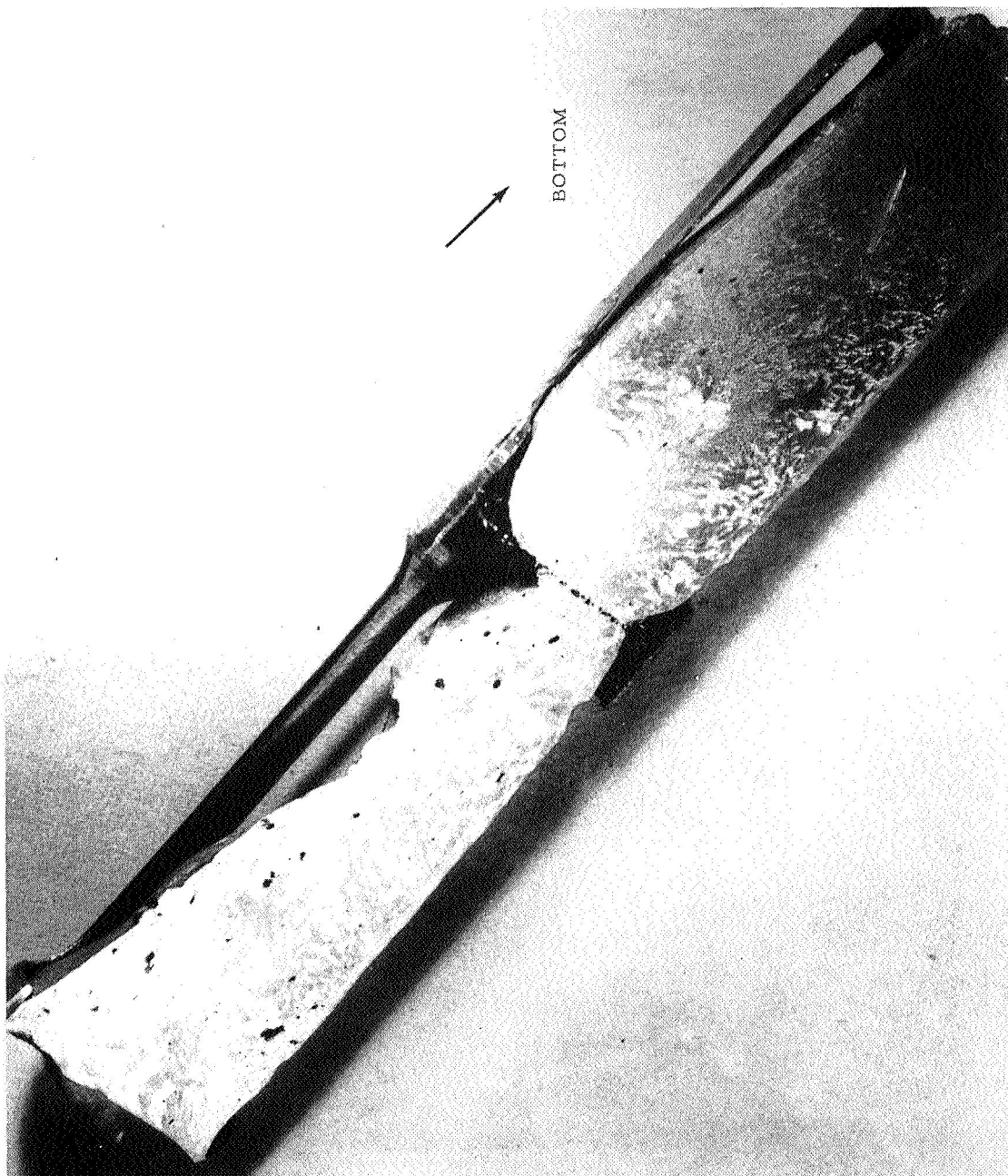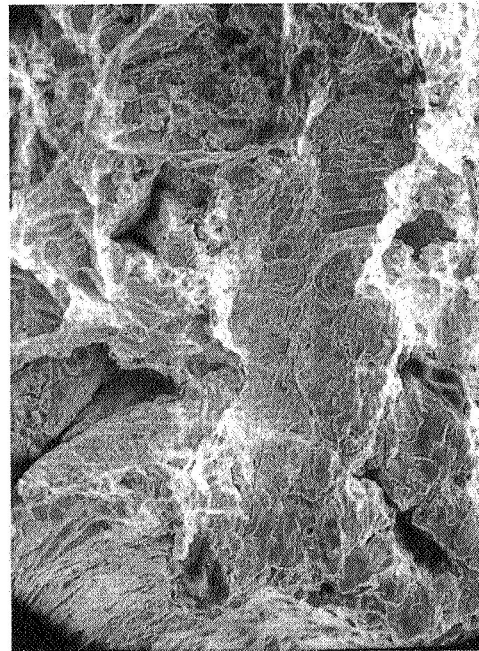As was seen from Sample 4, it seems that stabilizing dispersions, such as whiskers or an oxide layer formation on the bubble surfaces, are necessary processing requirements for metal foam in order to avoid bubble coalescence.

<u>Sample 8: 67 Percent In-Bi, 8 Percent Tungsten Particles, 25 Percent Argon.</u> The objective of this demonstration was to achieve a stable dispersion of dense spheres and argon gas bubbles in the metal matrix.

Radiographs of the unopened capsules (Fig. 26) show the differences in contact with the container. The flight sample was not in good contact with the walls of the container. The convex shape of the top surface of the control sample is misleading. This was probably due to loose tungsten spheres. The true solid surface was nearly flat.

It was determined that the argon gas had leaked from these samples during electron beam welding of the capsules.

Upon opening of the capsules many of the tungsten spheres fell out. The surfaces of both samples were covered with tungsten spheres.
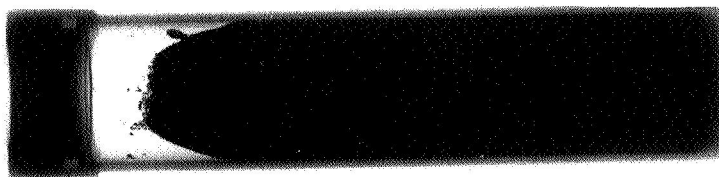
The samples were sectioned longitudinally and metallurgically prepared. The macrostructures (Fig. 27) show that there were few particles retained in the bulk of the control sample and a considerable number retained in the flight sample. Several "rosette" structures are also seen in the flight sample.

<u>Sample 10: 70 Percent In-Bi, 30 Percent Tungsten Particles.</u> The objective of this demonstration was to achieve a stable dispersion of the dense particles in the metal matrix.

When the capsules were opened, large quantities of tungsten particles fell out of the container. Figure 28 shows the control sample capsule partially opened. The holes in the sample contained large quantities of loose particles before opening. The surfaces of both control and flight samples were covered with particles.

The samples were sectioned longitudinally and prepared for metallography. The macrostructures (Fig. 29) show that there were few particles retained in the bulk of the control sample and a considerable number retained in the flight sample.

A photomicrograph, (Fig. 30) of the upper section of the flight sample shows the number of particles retained within the flight sample. The particles

44

Control   Flight
8C (Heat Sink End)   8F

Figure 27. Photomacrographs of longitudinal
sections; Samples 8C and 8F, etched, ~ 1.5X.
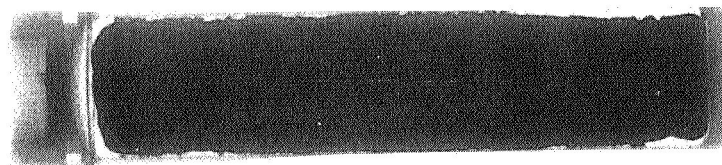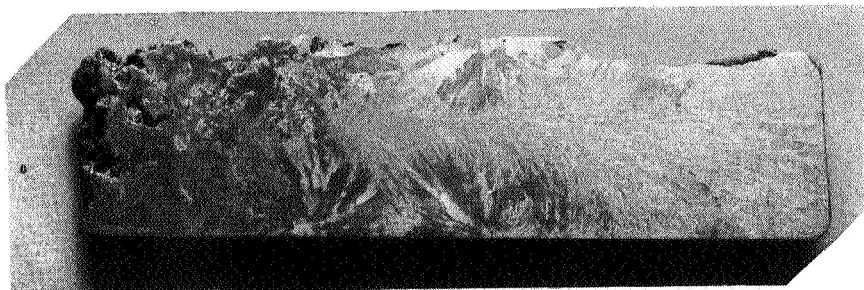
Control   Flight
8C   8F

Figure 26. X-ray radiographs, Samples 8C and 8F.

Control   (Heat Sink End)   Flight
10C   10F

Figure 29. Photomacrographs of longitudinal sections, Samples 10C and 10F.

Specimen No. 10C-00   Sample Configuration

                               X Complete
as filled   Segment
ground processed   Section
X flight processed

View: top   bottom
Side 0/90/180/270°

Enlargement: x
cm 1 2 3 4 5 6 7 8 9 10
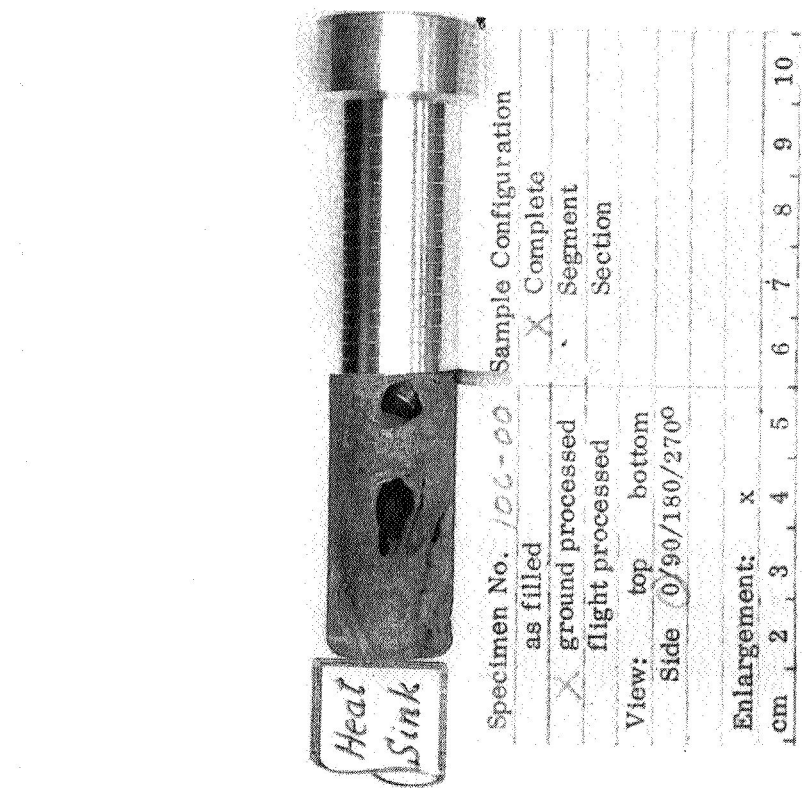
Figure 28. Sample 10C capsule partially opened.

46

were thought to be non-wetting. The flight sample was reheated and shaken several times because there had been RCS firings during the cooling cycle. This probably accounts for the increased number of particles in the bulk of the flight Sample 10 as compared to flight Sample 8.

Figure 30. Photomicrograph, upper section flight Sample 10F, (10X).

Sample 11. 68 Percent Paraffin, 7 Percent BeCu Fibers, 25 Percent Argon. The objective of this demonstration was to obtain a stable dispersion of fibers and gas in the matrix. Paraffin was chosen to give a visual display of the dispersion.

The sectioned flight and control samples are shown in Figure 31. There is practically no difference between the configuration of the flight and control sample. It is assumed that there was some acceleration applied to the flight sample after mixing which shifted the mass of fibers to one end of the capsule.

At the time of sample preparation, only Cerrobend-coated fibers were available. The coating melted during processing causing the fibers to solder themselves together. Consequently the objective of demonstrating fiber dispersion and fiber-bubble interaction was not achieved.

47
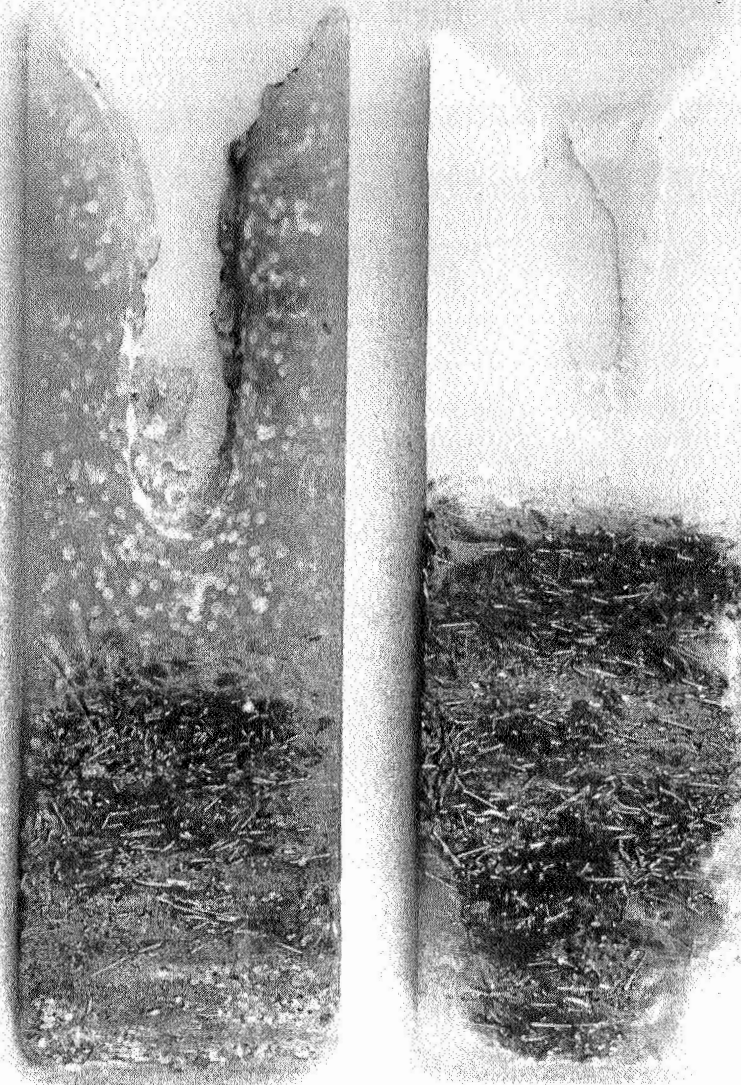
Flight   Control
11F   (Heat Sink End)   11C

Figure 31.   Photomacrographs of longitudinal
sections, Samples 11F and 11C.

48

One significant difference was the retention of gas bubbles in the paraffin in the flight sample in Figure 32. It was determined before opening the samples that most of the argon had leaked from the capsules and there was only a limited amount present. Therefore, only a low bubble density was obtained, and the problems of bubble coalescence and surface stabilization were eliminated. The stability of bubbles in liquid matrix under low-g solidification was, however, demonstrated.

Summary of Group B Results. Stable dispersions of particles and fibers were demonstrated in the flight samples even though some had non-wetting characteristics. A stable dispersion of gas bubbles was maintained in the whisker-reinforced flight Sample 4 and some dispersion of gas was maintained in flight Samples 7 and 11. The effectiveness of surface tension forces is evident in the rounded surface features of the flight samples which did not fill the capsules. This tendency to pull away from the container walls and hence to change the heat flow paths produced some unusual metallographic structures and suggests that free floating solidification experiments may be important to the understanding of the solidification process under low-g conditions.

Immiscible Materials Dispersions

The primary objective of this group of samples was to determine the effect of a low-g environment on the dispersion of immiscible materials. Three samples were prepared: one containing the immiscibles, sodium acetate trihydrate and paraffin only; one containing the immiscibles and an inert gas; and one containing the immiscibles and dense solid particles.

Processing of the samples was identical to that used for group B.

A comprehensive report on the characterization of the samples in this group and a comparison of the results with calculated data are given in Reference 5. Only a summary of those studies and results is given here.

Each of the samples was sectioned longitudinally and polished. The entire surface was photographed at 1X and 10X and selected areas at 50X. The sample halves were then sectioned, mounted, and polished, and additional photographs were taken of selected areas up to 1000X. Figure 33 is a schematic of the photographic documentation procedure.

Sample 6: 50 Percent Paraffin, 50 Percent Sodium Acetate. The sodium acetate and paraffin were segregated in the control specimen with the
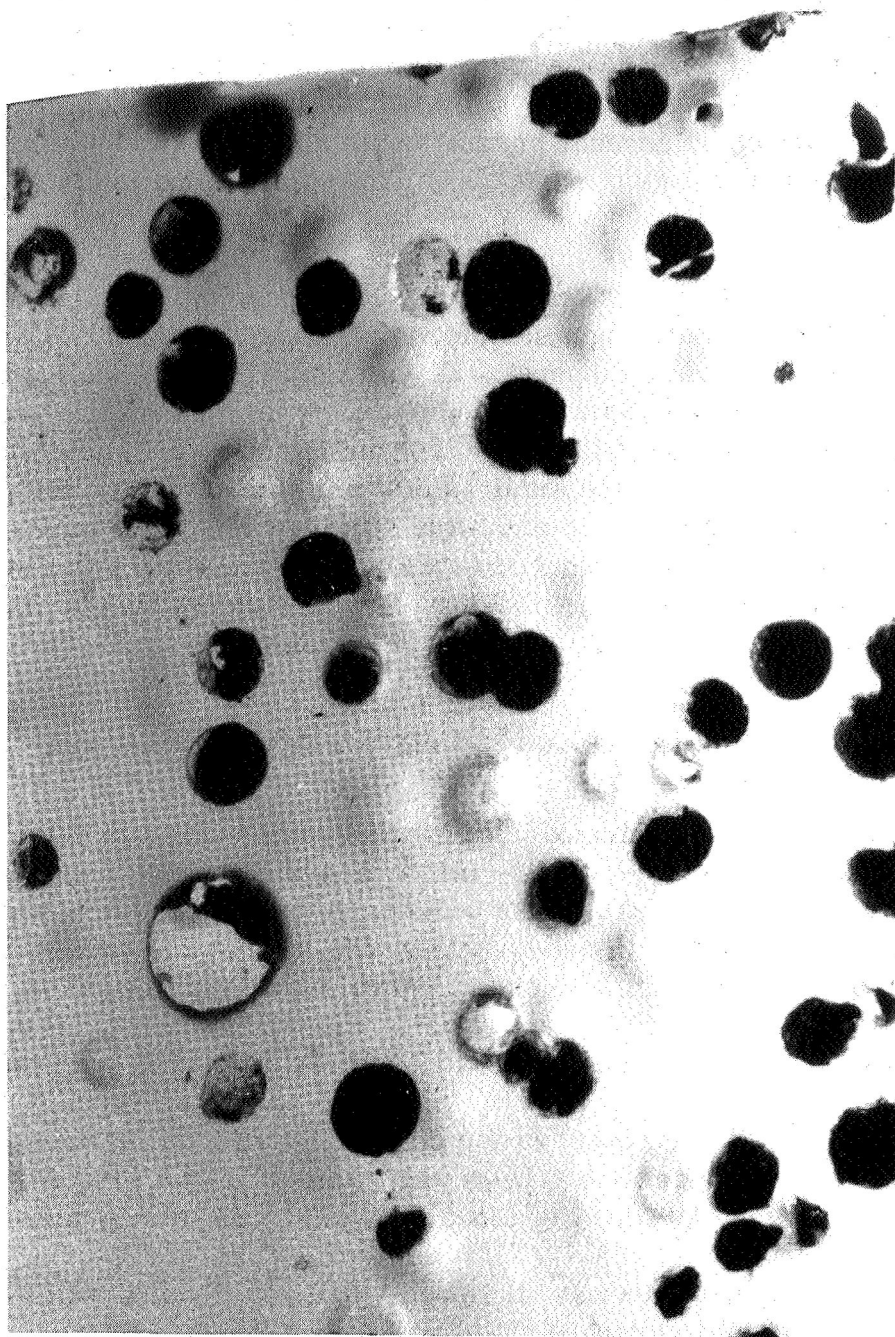
Figure 32. Close-up of bubble dispersion in flight Sample 11F.

50

1X PHOTOGRAPH

10X PHOTOMACROGRAPHS
(TOTAL SURFACE)

SELECTED
50X PHOTOMICROGRAPHS

SECTIONED

SURFACE PREPARATION
(ONE SIDE EACH SAMPLE FROM HEAT SINK END. TOP POLISHED IF APPLICABLE)

1X PHOTOGRAPHS

10X PHOTOMACROGRAPHS
(TOTAL SURFACE)

SELECTED 50X,100X,200X,500X,1000X PHOTOMICROGRAPHS

Figure 33. Schematic outline of photographic documentation procedure.

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exception that the shrinkage void in the paraffin was filled with sodium acetate (Fig. 34). The flight sample was partially segregated; however, there were visible some dispersions of sodium acetate in the paraffin at the top and paraffin in sodium acetate on the bottom.

Control   (Heat Sink End)   Flight
6C   6F
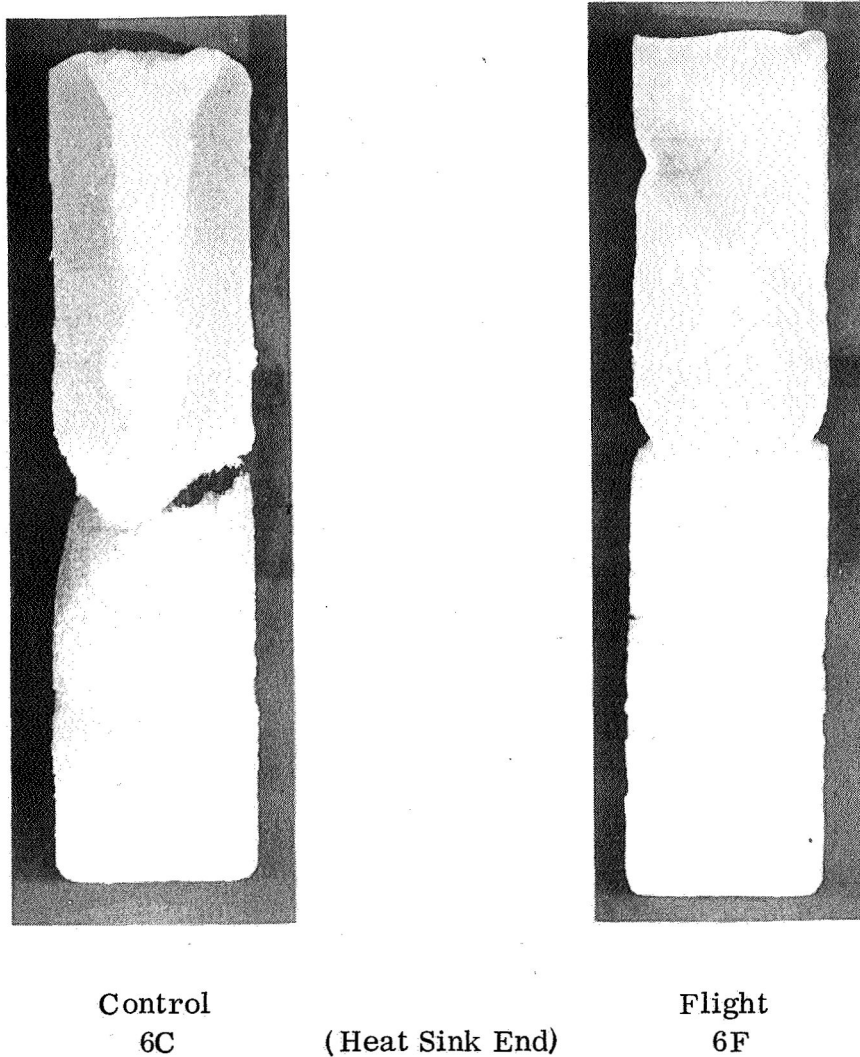
Figure 34. Longitudinal sections, Samples 6C and 6F.

Examination of the control sample at higher magnification determined that the paraffin was free of sodium acetate except for the filled shrinkage tube and that there was no paraffin dispersed in the sodium acetate. In the flight sample there were numerous paraffin inclusions dispersed throughout the sodium acetate and vice versa (Fig. 35). Photomicrographs at 500X and 1000X show

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Figure 35a. Flight Specimen 6F-A-00 (ctr), sodium acetate
dispersed in paraffin (50X).

Figure 35b. Flight Specimen 6F-A-00 (ctr), paraffin in
sodium acetate near heat sink (50X).

extremely fine dispersions of sodium acetate and paraffin (1 μ or less) 'in the grain boundaries of the sodium acetate (Fig. 36).

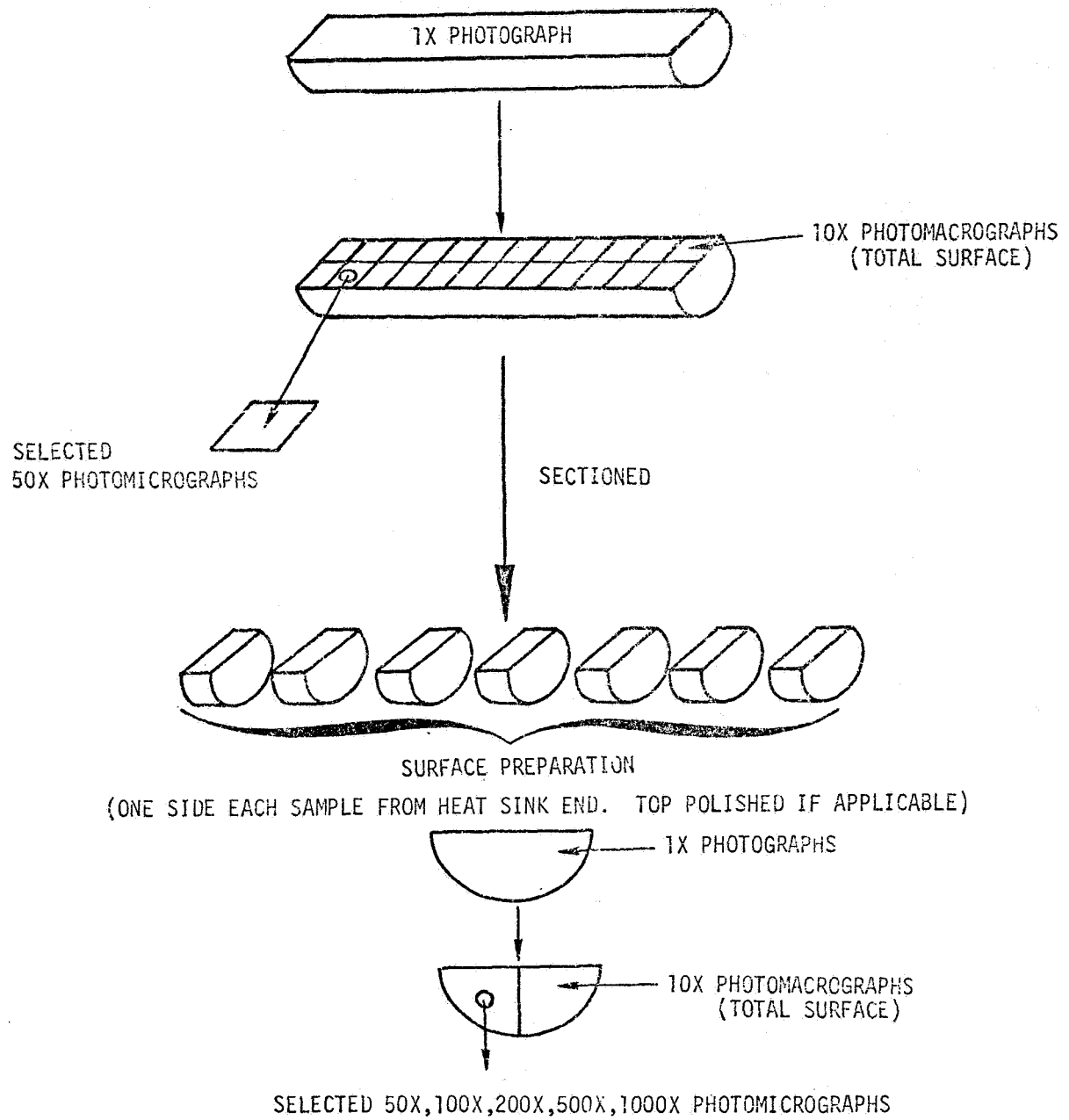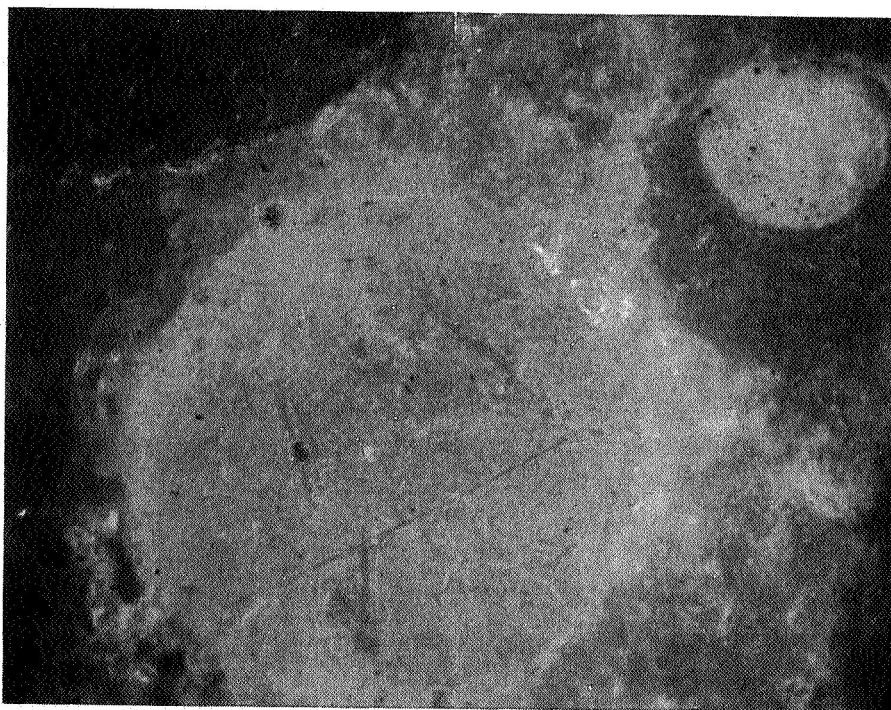Sample 9: 40 Percent Paraffin, 40 Percent Sodium Acetate, 20 Percent Argon. The sodium acetate and paraffin were completely segregated in the control sample and almost completely dispersed in the flight sample (Fig. 37). In the flight sample, the paraffin was primarily dispersed in the sodium acetate except at the top center of the sample where three large drops of paraffin coalesced and enclosed some sodium acetate spheres. Duplex dispersions (Fig. 38), consisting of a thin shell of paraffin surrounding a sphere of sodium acetate, were seen at various locations in the flight sample. Known also as the "grapeskin effect" such dispersions have been observed in oil-water mixtures and in wet jet fuel being pumped through pipelines. However, the effect has not been "frozen in" before. More complex dispersions of paraffin which surrounds the sodium acetate; which in turn surrounds paraffin, and the reverse, were also seen.

Examination of the control sample at higher magnifications determined that the sodium acetate and paraffin were completely segregated. In contrast, each sample surface of the flight sample showed dispersions of sodium acetate in paraffin or vice versa. The majority of the surfaces contained duplex and complex dispersions of sodium acetate and paraffin. Photomicrographs show dispersions of 1μ or less (Fig. 39).

Sample 12: 40 Percent Paraffin, 40 Percent Sodium Acetate, 20 Percent Tungsten Microspheres. The paraffin, sodium acetate, and tungsten microspheres were segregated in the control sample (Fig. 40) with the exception of a small cap of sodium acetate with a few tungsten microspheres over the top of the shrinkage tube. The cap was probably formed in the same manner as in control Sample 6.

In the flight samples there were three types of dispersions: a dispersion of paraffin (containing 90 percent of the microspheres) into sodium acetate; sodium acetate dispersed into paraffin; and a tungsten microsphere blend with a dispersion of sodium acetate. The tungsten microspheres were preferentially wetted by the paraffin; therefore, the continuous phase was paraffin with sodium acetate dispersed into it.

Examination of the control sample at higher magnification determined that the paraffin, sodium acetate, and tungsten microspheres were segregated except for slight dispersion at the interfaces.

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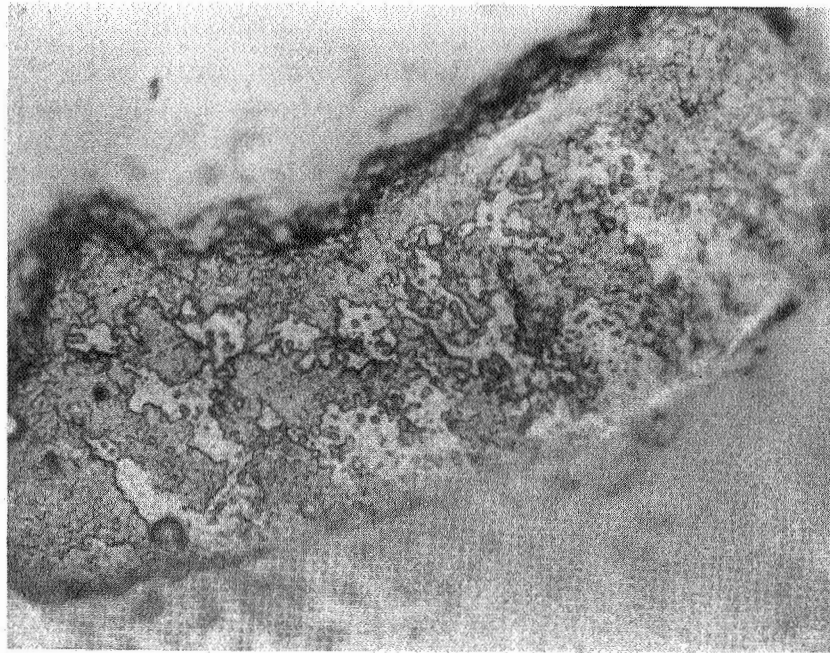
Flight Specimen 6F-A-05, 1μ dispersion primarily sodium acetate in paraffin (1000X).
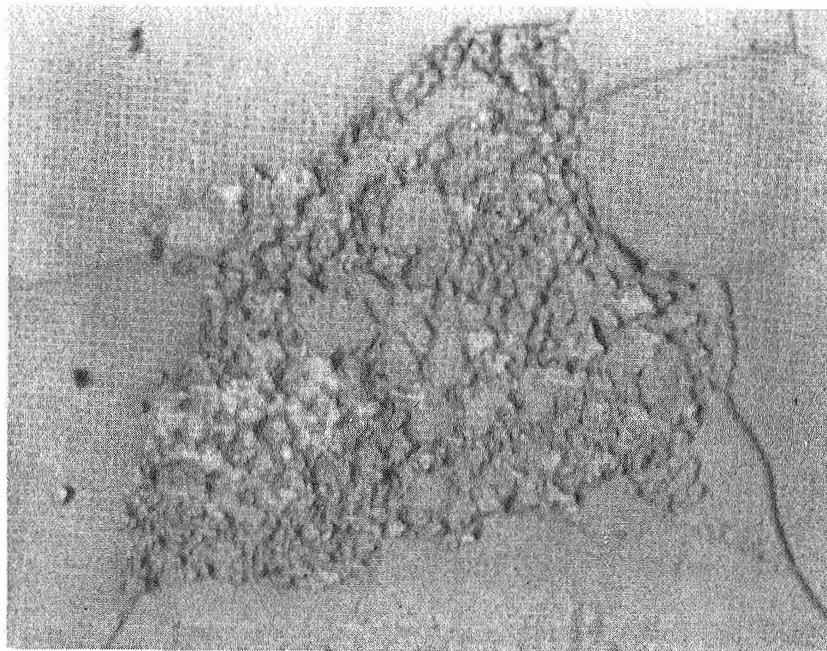
Flight Specimen 6F-A-02, 1μ dispersion of sodium acetate in paraffin, surrounded by sodium acetate (1000X).

Figure 36.   Fine dispersion in Specimen 6F.
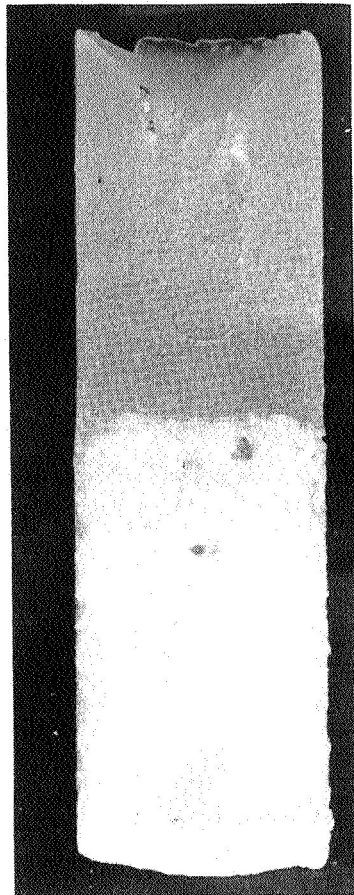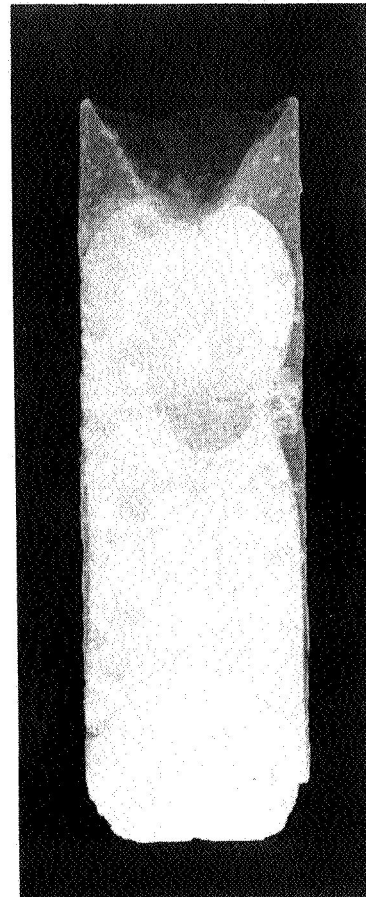
Control   Flight
9C   (Heat Sink End)   9F

Figure 37. Longitudinal sections, Samples 9C and 9F.
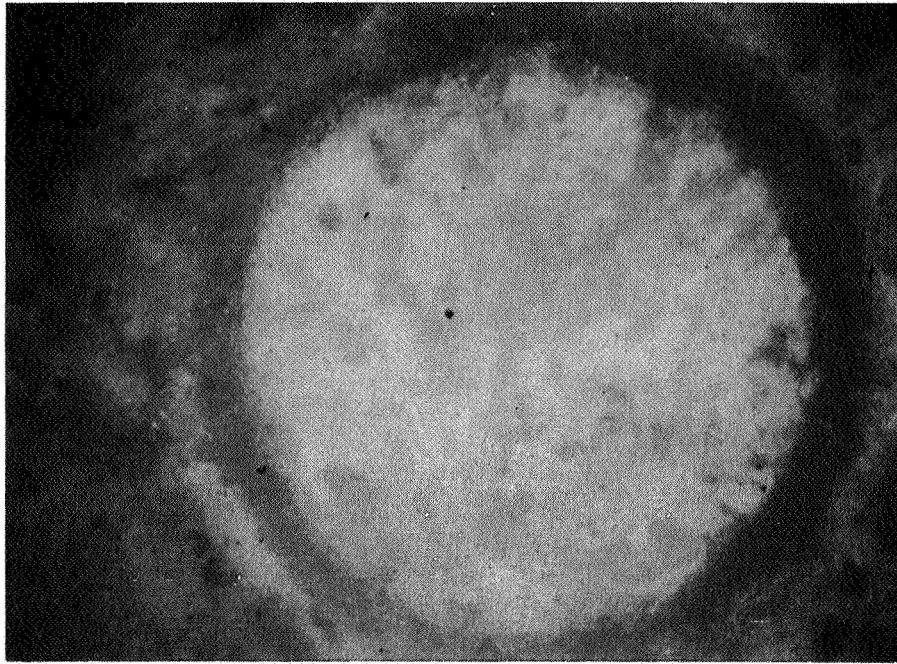
Flight Specimen 9F-A-00, duplex dispersion of paraffin-
sodium acetate in sodium acetate (50X).

Flight Specimen 9F-A-00, complex dispersion of paraffin-
sodium acetate paraffin in sodium acetate (50X).

Figure 38. Selected areas Specimen 9F-A-00.

Flight Specimen 9F-A-07 complex dispersions of paraffin-
sodium acetate-paraffin in sodium acetate, also sodium
acetate in paraffin in sodium acetate (50X).
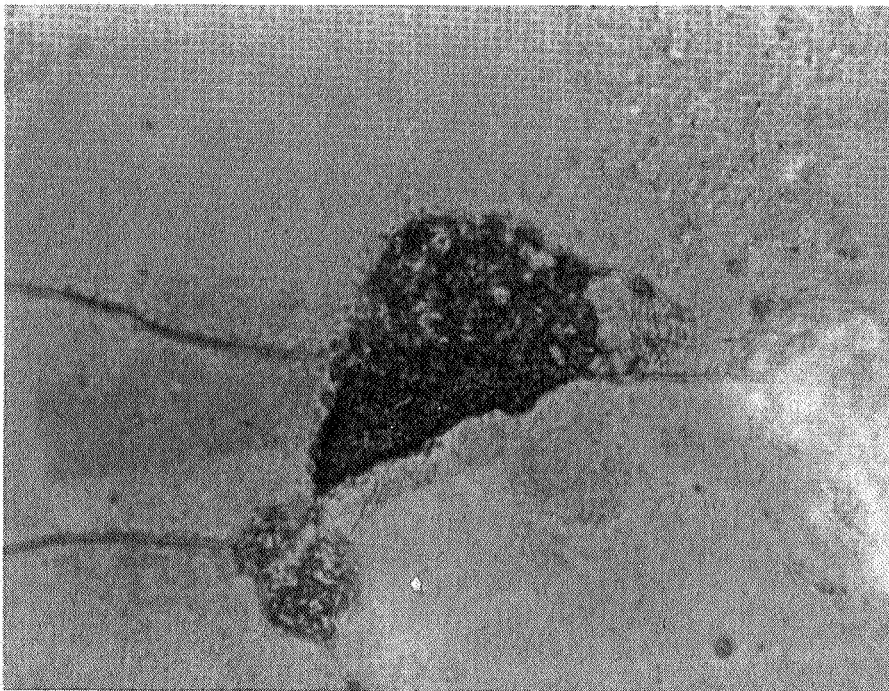
Flight Specimen 9F-A-01 1μ dispersion of sodium acetate in paraffin,
surrounded by sodium acetate, heat sink end (1000X).

Figure 39. Fine dispersions in Specimen 9F.
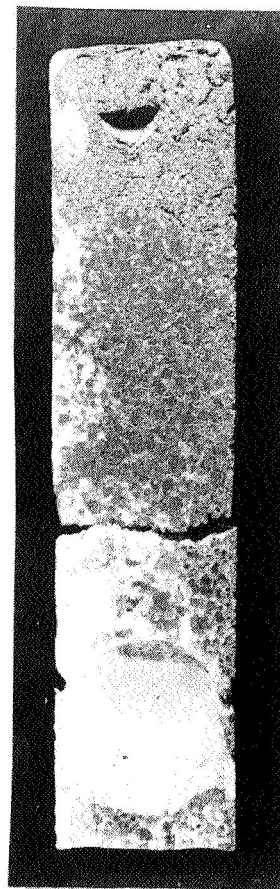
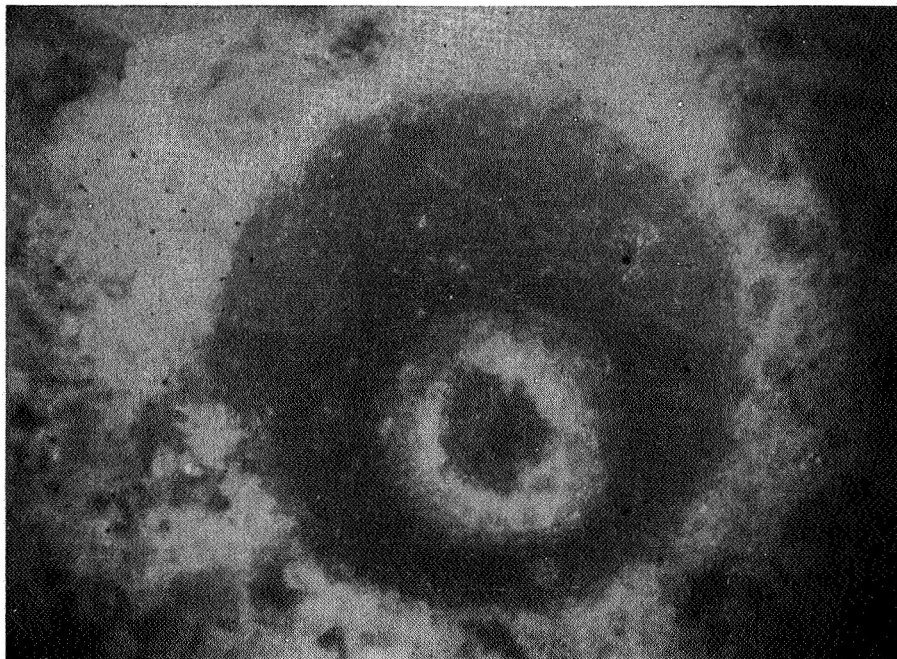Control   Flight
12C   (Heat Sink End)   12F
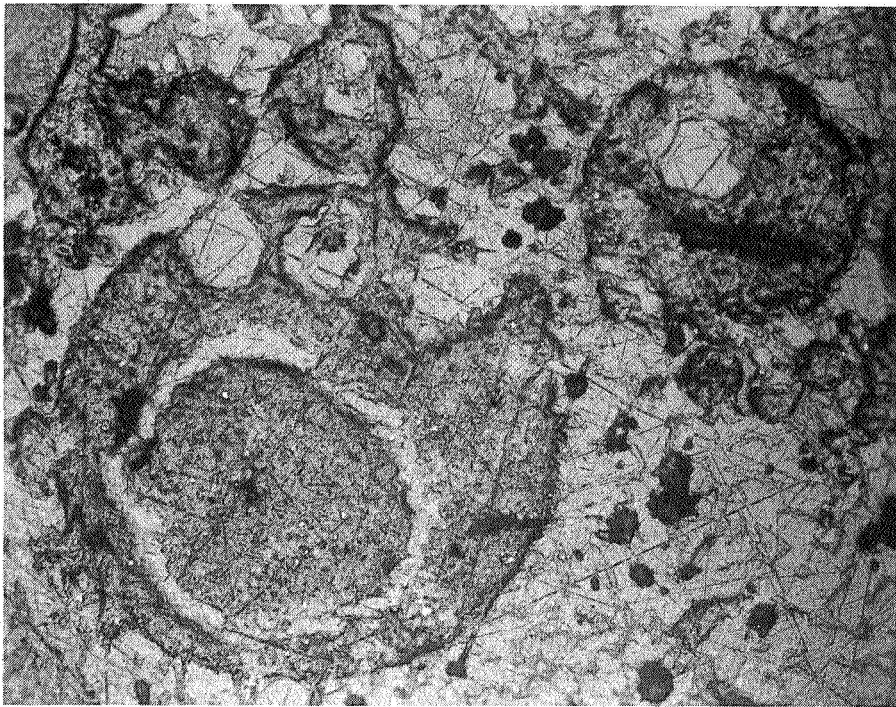
Figure 40. Longitudinal sections, Samples 12C and 12F.

In the flight sample three types of dispersions were found as mentioned above. Several areas containing dispersions of $1-\mu$-size particles were found although not as many as in flight Samples 6 and 9. This may have been due to the influence of the tungsten microspheres.

Comparison of Calculated Data with Demonstration Results. Theoretical analysis was performed by TRW to gain insight into the dominating phenomena, to predict various aspects of the thermal and mechanical histories of the · samples, and to compare these data with the demonstration results. Details of their analysis and comparison are given in Reference 5. The general conclusions from these studies were:

a. From the contact angle between the paraffin and the capsule wall on flight Samples 6 and 12, the acceleration level at solidification was approximately $10^{-3}g_e$ or 0.0098 M/sec^2.

b. Initial mixing was insufficient to thoroughly disperse the molten sodium acetate and paraffin.

c. The body force exerted on the specimens when they were inserted on the cooling pin had little or no effect on the distribution of the materials.

d. Since the cooling was essentially unidirectional, the surface-tension-driven convection forces were probably more predominant in causing fluid movement and droplet size and distribution differentiation than the acceleration forces present.

e. There is a possibility that some of the segregation of the materials in flight Sample 12 was caused by the Reaction Control System firings during capsule cool down.

f. From the calculated limiting velocity and sedimentation curves, the critical droplet diameter is approximately 100μ. Droplets less than this size tend to be stable in a thermal- or low-g gradient. This was partially corroborated by the droplet distribution measurements for paraffin.

Summary of Group C Results. Stable dispersions of immiscible materials processed in a low-g environment were demonstrated. The majority of the dispersions were between 25 μm and 1 mm in size, although areas containing dispersions of 1 μm or less and droplets up to 1 cm were observed in three flight samples. No droplets were observed in any of the control samples. Thus droplet formation is associated with mixing and solidification in low g.

Four stable types of dispersions were observed in the flight sample: sodium acetate in paraffin, paraffin in sodium acetate, a paraffin-tungsten microsphere blend in sodium acetate, and sodium acetate in a paraffin-tungsten microsphere blend.

Stable duplex dispersions and more complex forms of either sodium acetate in paraffin or vice versa were observed in all flight samples. Since this type of dispersion is unstable on earth, this was a direct demonstration of the unique potential for processing immiscible materials in a low-g environment.

None of the flight samples exhibited a homogeneous density distribution which would indicate complete dispersion. However, the axial gradation in density was more gradual in the flight samples and there were areas in each of relatively good dispersion. Flight Sample 9 exhibited the best dispersion.

The droplet size and distribution did not vary significantly in the radial direction, but droplet size did increase with distance longitudinally from the heat sink.

The comparison of observed properties with the theoretical analyses further established the feasibility of processing immiscible materials. These results can be applied to the design of improved experiments for development of technology applicable to processing immiscible materials in low g.

Conclusions

Detailed conclusions are given in each of the evaluation reports. Generally, it can be said that the low-g composite casting demonstrations served their purpose in that material structures were produced that cannot be duplicated on earth.

Where dispersants, such as fibers, particles, or gases had been added to the matrix, enhanced dispersion and distribution were found in the space processed samples.

Normally immiscible mixtures showed stable dispersions unattainable on earth.

Premixed samples made from 70 percent metal powders and 30 percent dispersed non-melting particles showed no really conclusive differences of

particle distribution between the flight and the control samples. Later investigations showed that the behavior on the ground of such dispersions is not well understood either.

The demonstration showed qualitative results in a very limited range of materials and under processing conditions which were not instrumented or highly controlled. Even so, the demonstrations were encouraging in that:

a. Unique material structures were produced.

b. New problems were raised which can be solved by future ground and flight experiments.

c. The importance of several factors which must be considered for process and experiment design were brought to light. These factors included the control of heating and cooling in low g when contact with heaters and heat sinks may be intermittent; control of nucleation and of mixing; and control of gases for distribution in the melt, or for removal from the melt and others mentioned in the text.

APPENDIX

INVESTIGATION OF GRAVITY INDUCED SEGREGATION EFFECTS IN PARTICLE DISPERSED COMPOSITES

Particle dispersed composite powder compacts of the type of Specimens 1 and 2 were kept in liquid state over long periods of time in order to investigate segregation effects.

Description of Specimens

Specimen V, available from the cancelled Apollo 15 demonstration, was utilized for this investigation. Three identical specimens were available, control Specimen V-C, flight Specimen V-F, and Spare V-S. Each specimen consisted of four segments which were separated by copper-coated aluminum discs. From top to bottom, segment 1 contained 20 percent B_4C particles in In-Bi powder matrix; segment 2, 10 percent B_4C in In-Bi powder matrix; segment 3, 20 percent W in In-Bi powder matrix; and segment 4, 10 percent W in In-Bi powder matrix.

All the powder compacts were prepared similarly to Specimens 1 and 2.

Test Procedure

The specimens were processed as follows:

Specimen V-C was processed in a vertical position in the laboratory using the Apollo 14 heater. The procedure was changed versus the original Apollo 14 operation insofar that the specimen was not melted in a horizontal position first and then put on the heat sink in a vertical position, but maintained in the vertical position in the heater during the cooling phase as well without using the heat sink. This extended the liquid phase of the sample to a total of 26 min. A portion of this specimen was reprocessed in the "Blue M" Mechanical Convection Oven through an additional 18-hr liquid phase. In order to establish the original condition as close as possible, the unused half segments were put back into a specimen container which was refilled with liquid In-Bi.

Specimen V-F was processed in vertical position in the Blue M Oven and held liquid for seven days.

Specimen V-S was processed as above and held liquid for seven days but in an upside down position.

Evaluation

The Specimens were opened and cut lengthwise similarly to the Apollo 14 specimens. One half of each segment was polished. The extreme difference in hardness between particles and matrix required careful selection of silicon carbide grinding papers in order to avoid particle relocation.

It is assumed that the visibility of the undisturbed matrix structure in (Figs. A-1 and A-2) indicates that relocation of particles did not occur during polishing.

The particle distribution was established through visual counting of sample areas on photographic enlargements of about 18X of the polished segment cross-sections.

The enlarged sample cross section was divided by a grid overlay into 2091 squares. Particle counts were made in 9 sample areas consisting of 36 squares each. Three areas; each in the top, center, and bottom sections, were counted. Thus an area of 324 grid squares out of a total of 2091 squares, or approximately 15.5 percent of the area, was evaluated.

Figure A-3 shows the average distributions of the top, center, and bottom areas of each of the four segments after different lengths of liquid processing time. Assuming that a variation of ± 15 percent from the average dispersion is typical for the powder compacting processes, it can be stated that no trend of change in distribution because of gravity-induced segregation can be observed.

The segments at the top and at the bottom showed the formation of a matrix area free from particles. In order to establish whether this is a gravity or a directional cooling effect, Specimen V-S was subjected to a seven-day

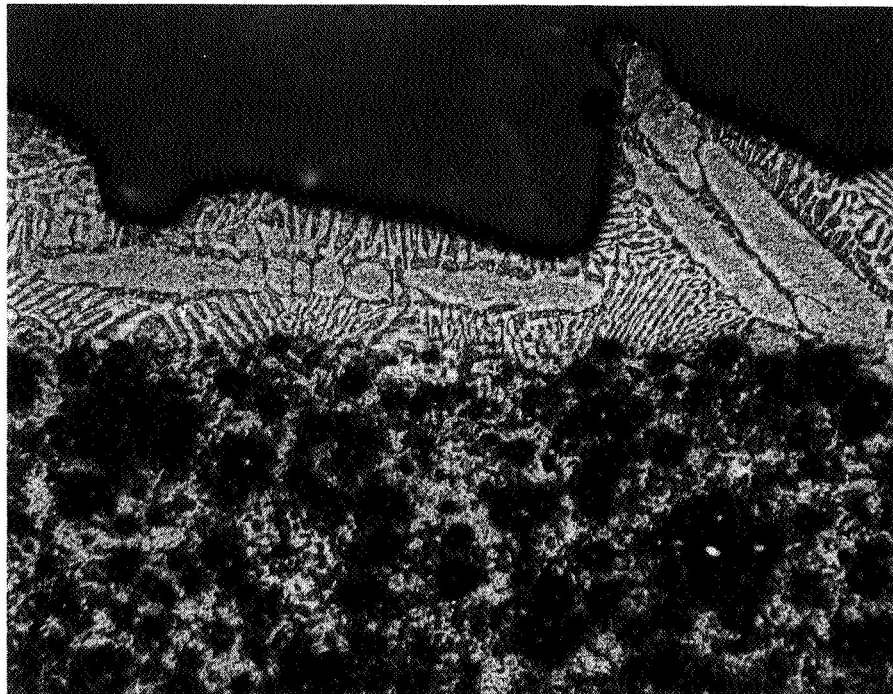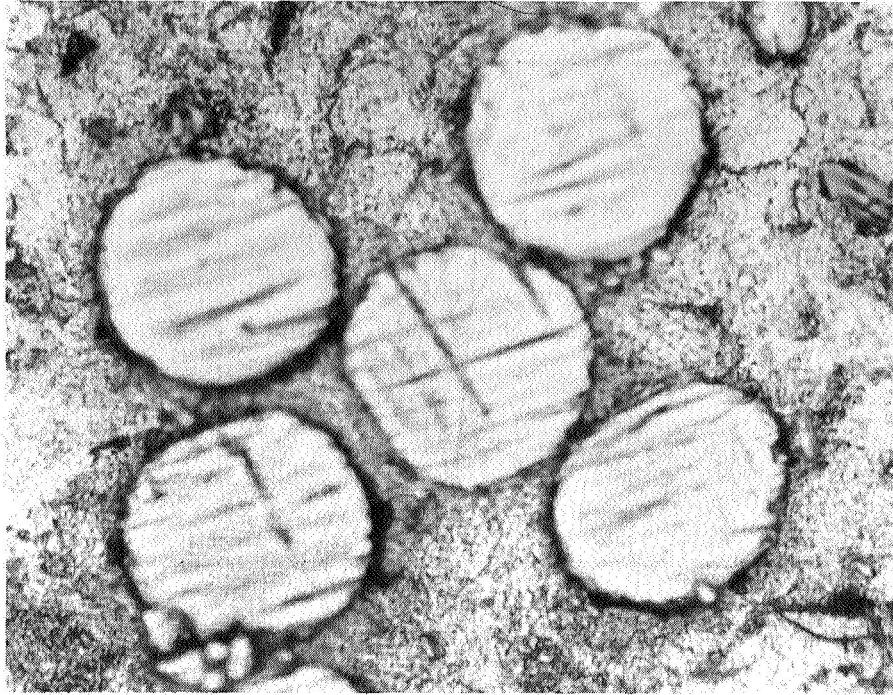Figure A-2. Segment 3 after 26 min, liquid phase processing W particles are successfully ground down with the matrix (650X).

Figure A-1. Upper edge of Segment 1 after 26 min liquid liquid phase processing, B$_4$C particles not attacked by polishing (100X).

Average Deviation
of 3 Top Areas

Average Deviation
of 3 Center Areas

Average Deviation
of 3 Bottom Areas

30   20   10   10   20   30

- 100 % +
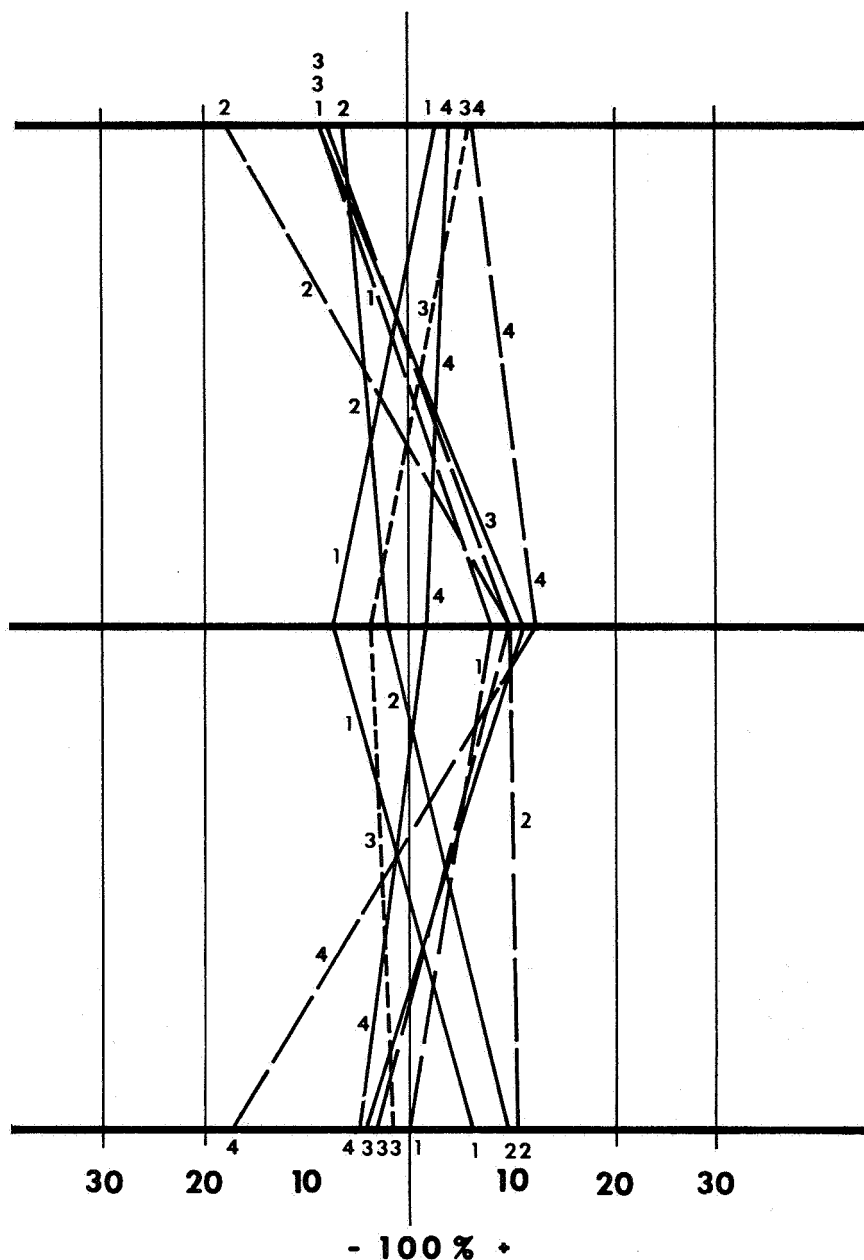
Deviation from Average Distribution

26 min Liquid   ————————
18 hr Liquid   ----------
7 days Liquid   ——— ———

Segments 1, 2, 3 and 4 are shown
by numbers.

Figure A-3. Average particle distribution.

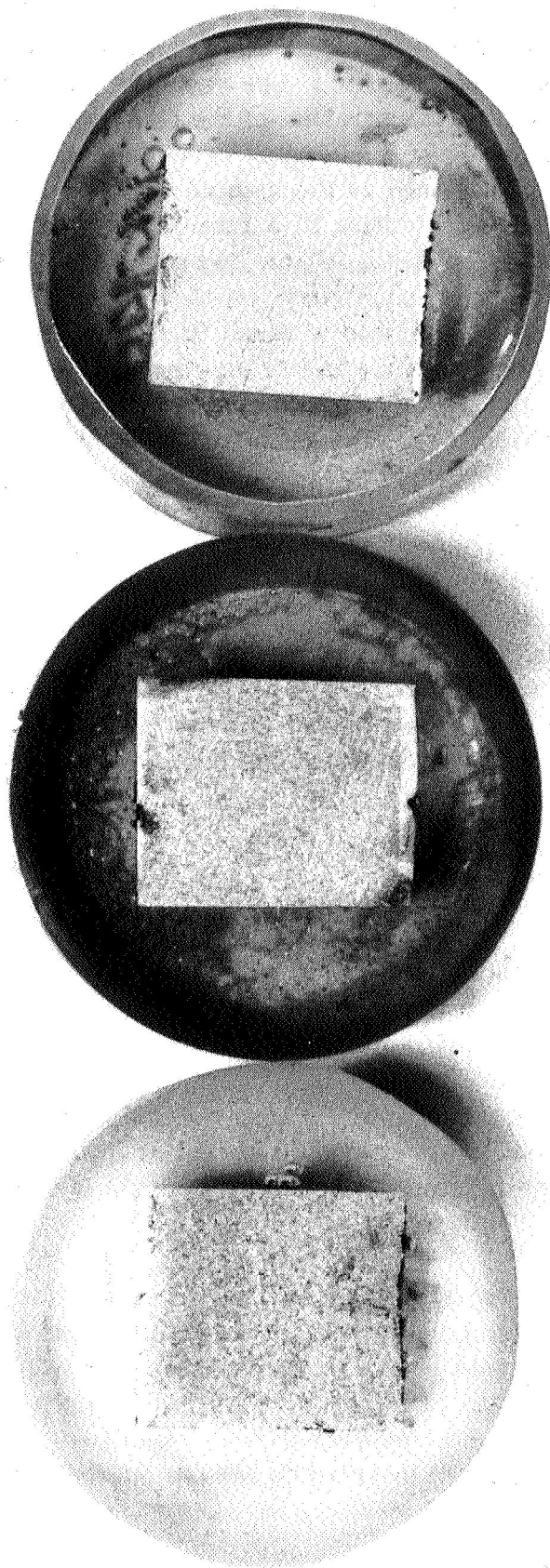liquid-phase processing in an upside down position. Figure A-4 shows the samples of segment 1 processed from left to right for 26 min, and 7 days in upright position and 7 days in upside down position. The result is that the particle-free areas appear independent of the duration of the liquid phase and always in the direction toward the top side of the capsule, from which obviously the solidification progresses toward the center of the sample. This rules out gravity segregation playing a role in the formation of particle free zones. It proves, on the other hand, that the matrix metal is able to move in between particles and that the boundaries of the original matrix powder do not form oxide barriers to the extent of preventing particle motion. If this would be the case, particles would have to be carried with the metal out-flow or, vice versa, could not have been carried with the progression of the solidification front. On the other hand, the latter is not verified by a higher particle distribution close to the clear metal zone.

Figure A-4 shows also that the porosity has been carried by the solidification front downward in the left and center sample. This means the gas transport occurred against the direction of gravity segregation. In the right sample, which was processed in upside down position, the porosity in the upper portion is more pronounced, which shows that the gas transport coincides with the direction of gravity segregation. The gas motion does not seem to influence the particle distribution, meaning no floatation effect has been observed.

Conclusion and Recommendation

The tests indicate that only minor gravity induced segregation could have occurred in the particle dispersed composites as used in Apollo 14 Specimens 1 and 2. This contradicts theoretical predictions stating that the tungsten particles, which are more than 2.5 times heavier than the matrix, should sink, and B_4C particles, which are more than 2.5 times lighter than the matrix, should rise. The viscosity of the In-Bi eutectic matrix metal at approx. 20° above melting temperature was experimentally verified by General Dynamics to be comparable to that of water.

Particles of similar size and shape and comparable density differential show gravity segregation in water under perfect wetting condition. The segregation-preventing mechanism, which must be present in the Specimen type 1 and 2 composites, is presently not known. One factor could be the presence of residual gas at the particle surface which causes the formation of free liquid surfaces which counteract particle motion. In order to investigate this factor,

After 26 min processing.   After 7 days liquid processing.   After 7 days liquid processing in an upside down position.

Figure A-4.   Segment 1, 20 percent B_4C in In–Bi.

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similar particle dispersions should be produced in a hard vacuum. Additional cleaning of the particle surfaces by ion bombardment or RF treatment in order to remove residual gas molecules is recommended. Subsequent segregation tests over extended liquid-phase processing time would show whether the above situation was caused by liquid-solid wetting imperfection because of the presence of gas.

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APOLLO 14 COMPOSITE CASTING DEMONSTRATION

By I. C. Yates

The information in this report has been reviewed for security classification. Review of any information concerning Department of Defence or Atomic Energy Commission programs has been made by the MSFC Security Classification Officer. This report, in its entirety, has been determined to be unclassified.

This document has also been reviewed and approved for technical accuracy.

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